

**QUALITY ASSURANCE PROJECT PLAN
Treatability Studies--Bench-Scale Testing**

**Northside Sanitary Landfill/ 151816
Environmental Conservation and
Chemical Corporation
Zionsville, Indiana**

**WA 07-5NH2.0 and 08-5N30.0
Contract No. 68-W8-0040**

MARCH 17, 1989



Engineers
Planners
Economists
Scientists

March 17, 1989

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Ms. Karen Vendl
U.S. Environmental Protection Agency,
Region V, 5HR-11
230 South Dearborn Street
Chicago, Illinois 60604

Subject: Northside Sanitary Landfill/Environmental
Conservation and Chemical Corporation, Final
Quality Assurance Project Plan, WA 07-5NH2.0 and
08-5N30.0

Dear Ms. Vendl:

We are pleased to submit 5 copies of the Final Quality
Assurance Project Plan, Treatability Studies--Bench-Scale
Testing, for Northside Sanitary Landfill/Environmental Con-
servation and Chemical Corporation.

Sincerely,

A handwritten signature in cursive script, appearing to read "Alpheus Sloan III".

Alpheus Sloan III
Site Manager

eam/GLT821/4
Enclosures

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Remedial Planning Activities
(ARCS V)
Contract Number 68-W8-0040
QUALITY ASSURANCE PROJECT PLAN (QAPP)

Project Title: Northside Sanitary Landfill/Environmental
Conservation and Chemical Corporation Sites
Zionsville, Indiana
Treatability Studies--Bench-Scale Testing

EPA WA Nos.: 07-5NH2.0, 08-5N30.0

EPA Project Officer: Doug Ballotti (Acting)

Prepared By: CH2M HILL

Date: 10/31/88

Approved


CH2M HILL Site Manager

Date: 10/31/88

Approved


CH2M HILL Program Manager


Date: 1 Nov 88

Approved


EPA Remedial Project Manager

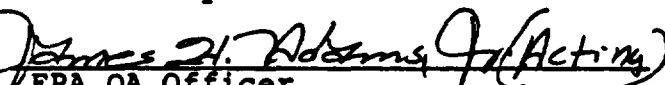
Date: 11/10/88

Reviewed


EPA Director, Central Regional
Laboratory

Date: 12/12/88

Approved


EPA QA Officer

Date: 12/12/88

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Section 1
PROJECT DESCRIPTION

INTRODUCTION

The United States Environmental Protection Agency (EPA) requires that all EPA contractors participate in a centrally managed quality assurance (QA) program. That requirement applies to all environmental monitoring and measurement efforts mandated or supported by the EPA. Each contractor generating data has the responsibility to implement minimum procedures to ensure that the precision, accuracy, completeness and representativeness of its data are known and documented. To ensure that the responsibility is met uniformly, each EPA contractor must prepare a written Quality Assurance Project Plan (QAPP) covering each project it is contracted to perform.

This QAPP presents the organization, objectives, functional activities and specific QA and quality control (QC) activities associated with bench-scale treatability studies for remedial design at the NSL/ECC site. Specifically, the portion of the predesign investigation addressed in this QAPP is the collection and analysis of groundwater and leachate to evaluate metal removal processes in bench-scale treatability studies (jar tests).

SITE DESCRIPTION

The ECC and NSL sites lie in a rural area of Boone County, Indiana, south of the intersection of State Route 32 and U.S. Highway 421 and about 10 miles northwest of Indianapolis. The ECC site occupies 6.5 acres immediately west of the 168-acre NSL site. The landfill occupies approximately 70 acres of the NSL site (Figure 1).

The area surrounding the sites is largely undeveloped. Land use to the east and south of the site is agricultural, and to the west and north it is residential. Approximately 50 residences are within 1 mile of the site.

An unnamed drainage ditch that separates the NSL site from the ECC site flows into Finley Creek near the southwest corner of the landfill. Finley Creek discharges into Eagle Creek about 1/2 mile downstream of the site. Eagle Creek then flows south for about 9 miles before emptying into the Eagle Creek Reservoir, which is used by the City of Indianapolis as part of its drinking water supply.

SITE HISTORY

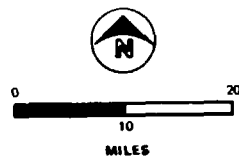
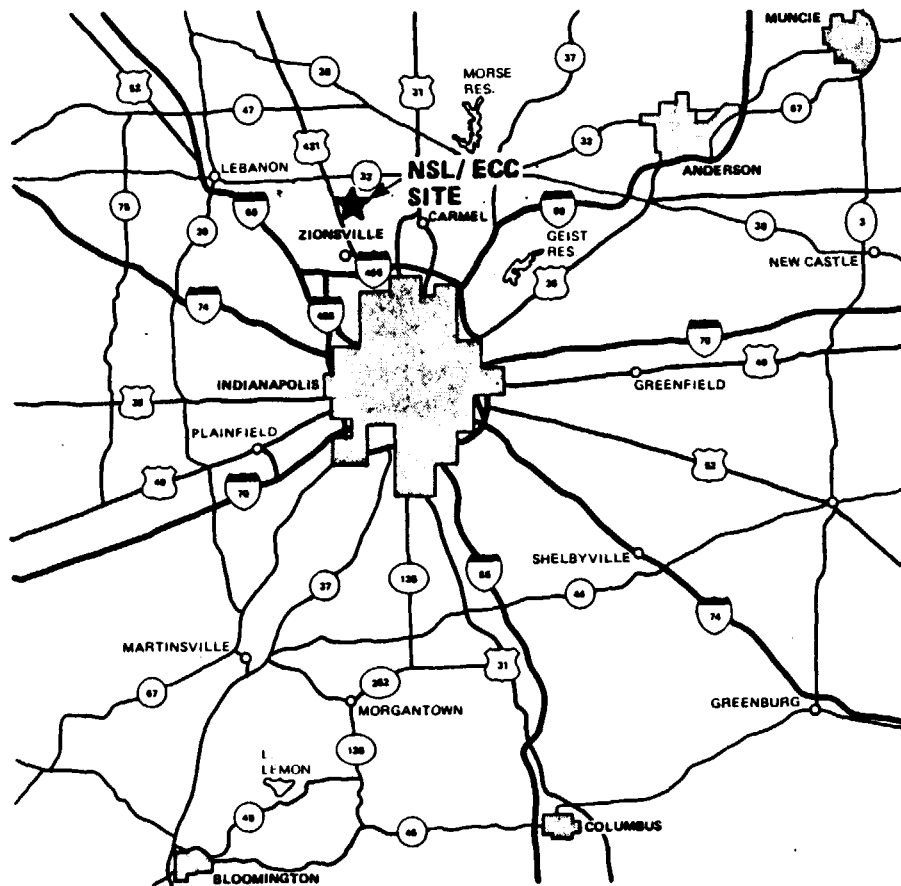
NSL is privately owned and operated as an active solid waste disposal facility. The landfill site has been active since at least 1962 and has accepted various industrial and municipal wastes during the course of its operation. The vice president of NSL has estimated that 16 million gallons of hazardous waste have been disposed of in the landfill. A 3-acre oil separation lagoon is visible in a 1977 aerial photograph. The site has had recurring operational deficiencies as reported by the Indiana State Board of Health (ISBH). The U.S. EPA detected leachate running into Finley Creek, and groundwater contamination was detected in monitoring wells at the site. The site was placed on the National Priorities List (NPL) in 1983.

ECC began operation in 1977 and was engaged in the recovery, reclamation, and brokering of primary solvents, oils, and other wastes received from industrial clients. Waste products were received in drums and bulk tankers and prepared for subsequent reclamation or disposal. Reclamation processes included distillation, evaporation, and fractionation to reclaim solvents and oil. ECC wastes disposed of at NSL reportedly included 5,000 gallons/month of waste fluids from the oil reclamation, still bottoms and solvent recovery waste, 50 to 80 drums/day of paint sludge, thinner, stain, and resin sludge, and at least 7,000 drums whose contents are unreported.

Drum shipments to ECC were halted in February 1982 after U.S. EPA and ISBH investigations found evidence of accumulated contaminated stormwater onsite, inadequate management of drum inventory, and several spills. In 1983, ECC was placed on the NPL. The EPA subsequently removed, treated, and disposed of cooling pond waters, about 30,000 drums of waste, 220,000 gallons of hazardous waste from tanks, and 5,650 cubic yards of contaminated soil and cooling pond sludge.

Remedial investigations of soil, hydrogeology, surface water, and sediment at both sites began in 1983 and continued to November 1985. Details of the investigations are included in the ECC and NSL Remedial Investigation (RI) reports. Data for groundwater and leachate samples from the RI reports are found in Appendix B.

Soil contaminants found onsite at the ECC site were primarily volatile organic compounds (VOCs) and phthalates. VOCs in the surface soils have migrated downward. A shallow sand and gravel deposit (approximately 18 feet below ground surface) has also been found to be contaminated with VOCs, though



LEGEND

- NSL SITE
- ECC SITE
- LANDFILL AREA

SOURCE: U.S.G.S. 7.5 min. quadrangle, Rossion, Ind. 1988.

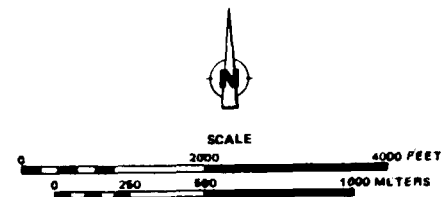
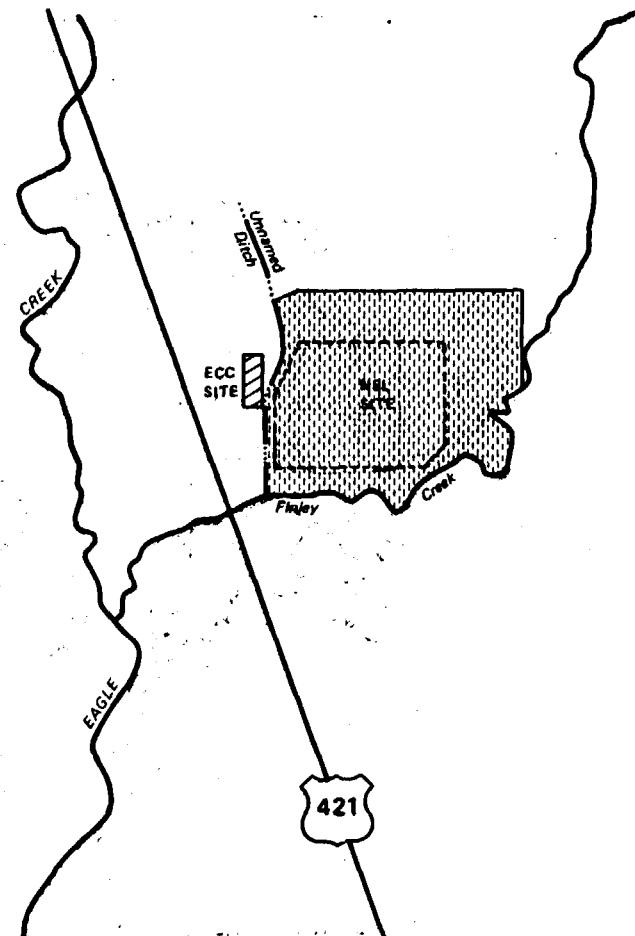


FIGURE 1
LOCATION MAP
NSL/ECC QAPP

the source may be a former onsite cooling pond rather than downward migration from the surface soils. Organic contaminants were also found in Finley Creek downstream of the site.

Soil contaminants detected in peripheral subsurface soils at the NSL site were primarily semivolatile organic compounds and some VOCs, found at depths of 13 to 15 feet. A sand and gravel lens at or near the ground surface in the southwest corner of the site was found to be contaminated with VOCs. PAHs and VOCs were also detected in Finley Creek downstream of the site.

The Feasibility Study reports for the NSL and ECC sites (dated December 5, 1986) contain more detailed information on the nature of site contamination and site hazards. The alternative recommended for site remediation includes groundwater and leachate collection and treatment.

Predesign and supplemental investigations including geotechnical, hydrogeologic, and groundwater, leachate and soil sampling were conducted in August 1987 and the spring of 1988. Details of these investigations are found in Predesign Technical Memorandums 1 and 2 (August and November 1988) and data from the groundwater and leachate samples are included in Appendix B.

TARGET COMPOUNDS

The compounds of major concern at the NSL/ECC site are TCL metals, VOCs, and semivolatile organic compounds in soil and groundwater. Sample analyses for the bench-scale metal precipitation study will focus primarily on the TCL metals and VOCs. The bench-scale precipitation study is to assess the removal of metals in the coagulation/flocculation process. Conventional pollutants such as BOD₅ and suspended solids will also be affected. It is not expected that precipitation processes will alter the concentrations of organic compounds in the influent, although there may be some release of VOCs during mixing. Semivolatile organic compounds should not be effected by the precipitation process. Samples will be analyzed using Routine Analytical Services (RAS) protocols and, for copper and chromium, Special Analytical Services (SAS) of the Contract Laboratory Program (CLP). Conventional water quality parameters, chromium, and arsenic will be analyzed by the ISBH laboratory. Required quantification limits for target compounds are listed in Appendix A.

The ISBH laboratory will perform the groundwater and leachate sample analysis for conventional water quality parameters, chromium and arsenic because:

- o The analyses performed by the laboratory count toward the state's participation in the Superfund program.
- o Quick turnaround of sample analytical results can be obtained.
- o CH2M HILL has previously had good experience with the ISBH laboratory for similar analyses.
- o Use of the ISBH laboratory saves costs that would otherwise be charged to the CLP.
- o The laboratory location is convenient, reducing the possibility of problems associated with sample storage and management.

The monitoring system for ISBH protocols and capabilities has been set up and the EPA audits the laboratory annually through the NPDES program. The ISBH laboratory is subject to audits for the Superfund project.

PROJECT OBJECTIVES

The objectives of the bench- and pilot-scale treatability studies are to collect data needed to design and implement the EPA preferred alternative, which consists of:

- o Deed and access restrictions
- o RCRA cap over the site
- o Rerouting of surface water
- o Leachate collection and treatment
- o Groundwater collection and treatment
- o Monitoring

Details of the selected remedial alternative are presented in the Record of Decision (ROD) for the site (September 25, 1987).

The bench-scale treatability study will address only the evaluation of groundwater and leachate treatment. This QAPP is concerned with the collection of groundwater and leachate for use in bench-scale treatability studies to evaluate metal removal including chemical precipitation requirements. The second phase of the study, not addressed in the QAPP, will

evaluate subsequent treatment processes for removal of conventional pollutants and organic compounds.

Leachate and groundwater samples will be collected to obtain data for the bench-scale treatability study. Leachate samples will be taken from an onsite leachate tank and groundwater from existing wells along the south-southwest perimeter of the site and in the supplemental investigation area. Well locations lie along the general alignment of the proposed groundwater collection system or extraction well field. CLP and ISBH data will be used to assess the removal of metals and the effect of metal removal on residual VOCs and conventional pollutants from optimized precipitation processes. Pilot study design and treatment plant design will be developed from the results.

The actual bench-scale tests will be contracted to an outside laboratory. The contracted laboratory will be responsible for sample analysis during the evaluation of different treatment processes. Characterization of the influent for use in the bench-scale studies and determination of concentration in the final effluent from the treatment process as optimized by the contractor will be determined through analysis by the EPA and ISBH. TCL metals and VOCs will be analyzed through the CLP. The ISBH will perform conventional pollutant, chromium and arsenic analysis.

The sampling team will perform field analysis of pH, temperature, and specific conductance. HNu or OVA air monitoring for VOCs will be used to monitor the safety of sampling personnel.

PROJECT TASKS

Groundwater Sampling

The pump test well and ECCMW17, which were installed during the supplemental investigation, will be sampled for laboratory and bench-scale testing. At least five well volumes of water will be purged from the well using a peristaltic pump, bailer, or a submersible pump before samples are taken. Samples will be analyzed in the field for pH, temperature, and specific conductance. Unfiltered samples will be sent to CLP laboratories for metals and VOC analysis. The ISBH laboratory will analyze the unfiltered samples for biochemical oxygen demand (BOD), chemical oxygen demand (COD), total suspended solids (TSS), volatile suspended solids (VSS), total dissolved solids (TDS), chlorides, sulfates, nitrate, nitrite, alkalinity, ammonia nitrogen,

total Kjeldahl nitrogen (TKN), total chromium, hexavalent chromium, arsenic, and total phosphorus.

VOC samples will be collected using a dedicated Teflon or stainless steel bailer. Approximately one 55-gallon drum of sample from each location will be collected for use in the bench-scale testing. Effluent from the laboratory-optimized metal treatment process will also be sent to the CLP laboratories to be analyzed for metals and VOCs and to the ISBH laboratory for conventional pollutants. The groundwater sampling procedures are described in the Site Sampling Plan (Appendix A). A total of twenty groundwater samples (plus replicates and blanks) will be submitted for analysis. Ten groundwater samples (plus replicates and blanks) will be collected in the field and submitted for analysis. Ten groundwater samples (plus replicates and blanks) from the optimized treatment process will be submitted for analysis.

Leachate Tank Sampling

Samples will be taken from leachate collection tank No. 2 for bench-scale testing and laboratory analysis. The samples will be obtained using a diaphragm pump equipped with flexible tygon tubing.

Leachate samples will be submitted for analysis in the same manner as groundwater samples. VOC samples will be collected from the observational pumpout port of the tank using a stainless steel or teflon bailer. Monitoring for released VOCs using an OVA or HNu is recommended before tank sampling for the safety of sampling personnel. Approximately one 55-gallon drum of leachate will be collected for use in the bench-scale study. Effluent from the laboratory optimized treatment process for leachate will be submitted for analysis in the same manner as groundwater samples.

Sampling procedures are described in the Site Sampling Plan. A total of ten tank samples (plus replicates and blanks) will be submitted for analysis. Five leachate samples (plus replicates and blanks) will be collected in the field and submitted for analysis. Five leachate samples (plus replicates and blanks) from the optimized treatment process will be submitted for analysis. Sampling locations for both groundwater and leachate are shown in Figure 2.

Treatability Study and Bench-Scale Testing

The contractor, given sufficient amount and characterization of influent, will be required to optimize the precipitation

process to achieve the highest percentage removal of the target metals. For example, precipitation by hydroxide or sulfide or coprecipitation could be examined. The contractor will determine the amount of influent water required to perform these tests. Specification in regards to documentation and analysis to be required of the contractor will be presented in the bid document.

Data Needs and Data Quality Objectives

The results from bench-scale testing will be used to evaluate the effectiveness of an optimized metal precipitation, coagulation, and flocculation process and to design pilot studies for evaluating downstream treatment processes. Data needs include:

- o Characterization of the effectiveness of the metal precipitation process
- o Chemical characterization of the influent before treatment by metal precipitation
- o Chemical characterization of the effluent from the optimized metal precipitation process

Data quality objectives for the metal precipitation bench-scale tests will be to:

- o Determine whether the optimized metal precipitation processes can treat the leachate and groundwater from the site
- o Determine which volatile organic compounds and conventional pollutants will be removed in the metal precipitation process to design the pilot tests and downstream treatment processes
- o Determine if the proposed NPDES permit limits (Appendix G) can be met by the metal precipitation processes
- o Screening of VOC concentrations with an HNu or OVA to monitor the safety of field sampling personnel and select proper personal protective equipment as outlined in the site safety plan
- o Identify characteristics of the influent which will affect precipitation processes with field

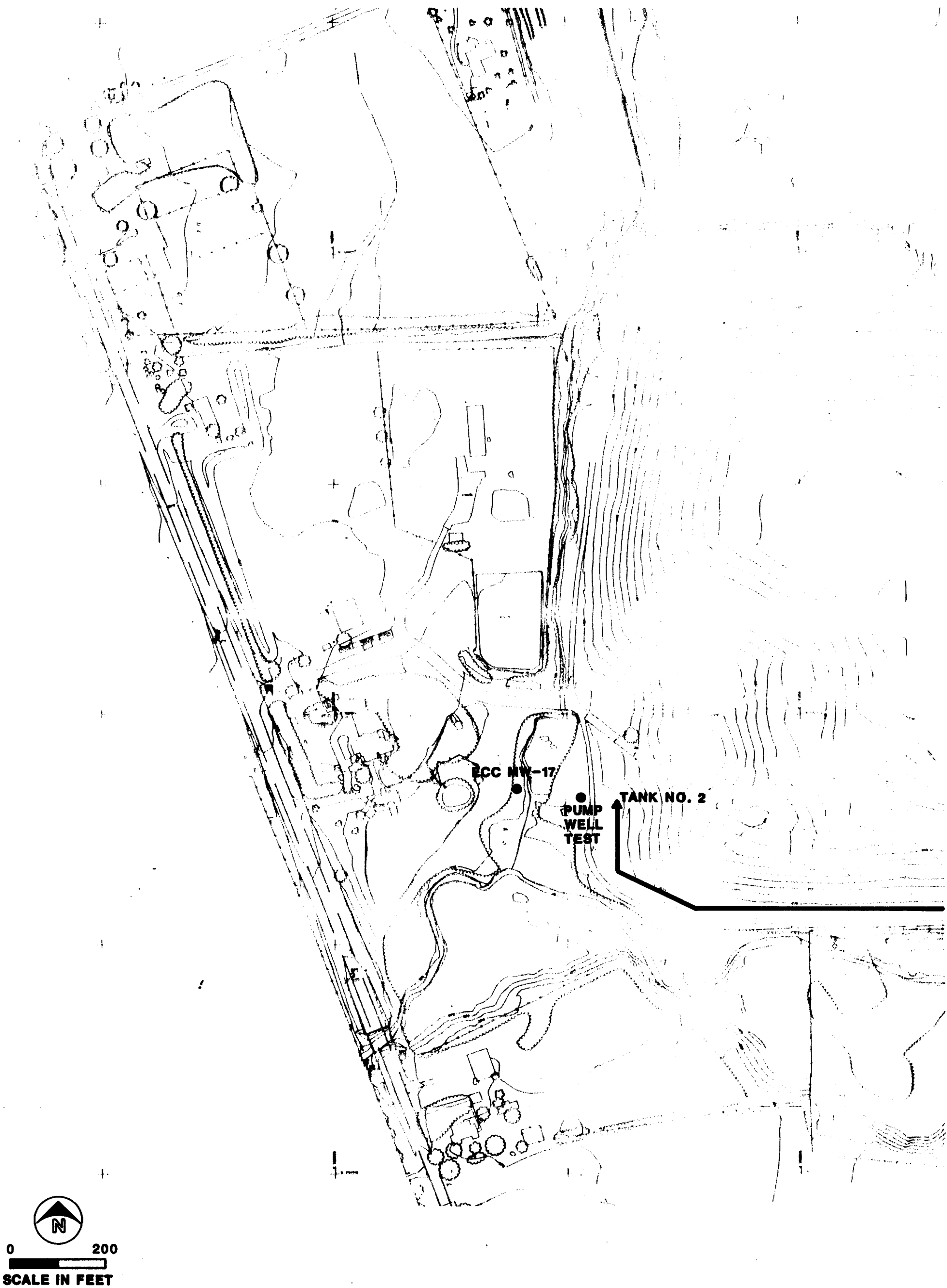


FIGURE 2
SAMPLING LOCATIONS
NSL/ECC



measurements, including temperature, specific conductivity, and pH

SAMPLE NETWORK AND RATIONALE

Table 1 summarizes anticipated analyses to be performed on samples from the NSL/ECC site. The compounds to be analyzed for during the bench-scale treatability study are listed in Table 1. Sampling methodologies and protocols are described in the Site Sampling Plan (Appendix A).

PROJECT SCHEDULE

The sampling schedule has not yet been determined. Bench-scale testing will proceed as soon as the contracted laboratory receives the collected samples. Figure 3 shows the proposed schedule and duration of the proposed tasks.

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Table 1
SAMPLING AND ANALYSIS SUMMARY
NSL/ECC SITE

[illegible]

[illegible]

- NOTES:
1. See Table A-2 for a complete list of parameters.
 2. See SAS request forms in Appendix C.
 3. See ISBH protocols in Appendix D.
 4. Field replicate samples will be collected at a frequency of one per group of ten or fewer investigative samples.
 5. Field blank samples will be collected at a frequency of one per group of ten or fewer investigative samples.
 6. Trip blank samples are not included in the matrix total.
 7. A trip blank sample will be shipped with each shipping cooler of water samples for VOC analysis.
 8. Matrix spike and matrix spike duplicates are not included in the total number of samples.
 9. For samples designated for matrix spike and matrix spike duplicate analysis, extra volumes of samples will be collected. Triple the normal volumes of samples will be collected for VOC analysis, and double the normal volumes for extractables, pesticides/PCBs.

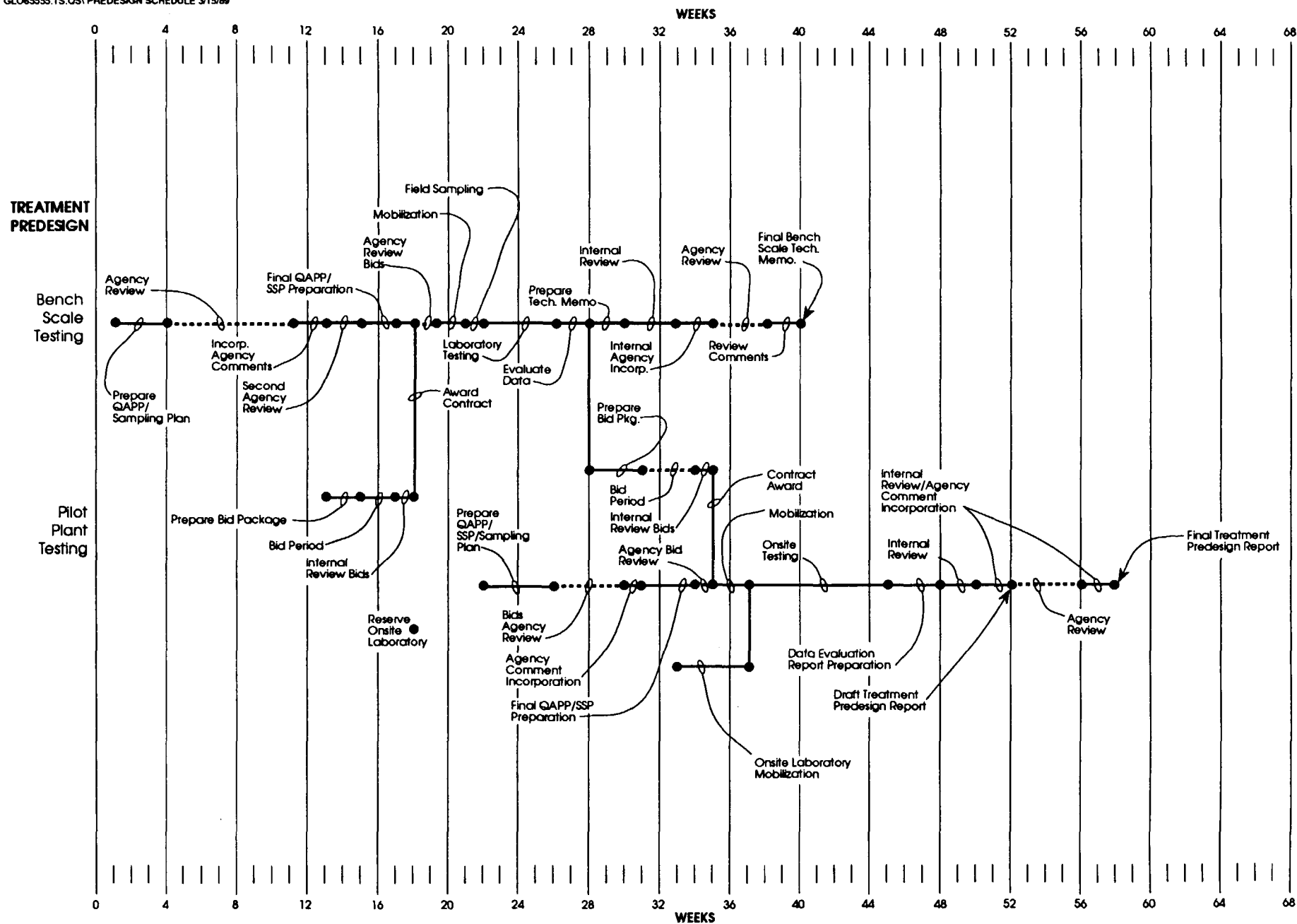


FIGURE 3
PREDESIGN SCHEDULE
NSL/ECC QAPP

Section 2
PROJECT ORGANIZATION AND RESPONSIBILITY

CH2M HILL has overall responsibility for all phases of the bench-scale treatability study, including overall management and QA/QC. CH2M HILL will perform the field sample collection, field measurement, and field screening and prepare the study report.

TASK PM--PROJECT MANAGEMENT

Project management will be conducted through CH2M HILL's regional office in Milwaukee. Contact will be maintained with the EPA's Remedial Project Manager (RPM) during all phases of the project.

Monthly reports will be submitted to keep the EPA apprised of the technical, financial, and schedule status of the project. Other CH2M HILL responsibilities include controlling budgets and schedules; selecting, coordinating, and scheduling staff and subcontractors for task assignments; and maintaining project quality control and assurance programs.

TASK QC--QUALITY CONTROL

Throughout the project, a quality control review team will periodically review project files, project deliverables, and site inspections. The team will consist of three professionals with experience from appropriate disciplines related to the problems and investigations at the site.

The following responsibilities have been assigned to the project:

- o Remedial Project Manager (RPM)
Karen Vendl (U.S. EPA)
- o Site Manager (SM)
Alpheus Sloan III (CH2M HILL)
- o Program Manager (PM)
John Fleissner (CH2M HILL)
- o Quality Assurance Manager (QAM)
Greg Peterson (CH2M HILL)
- o CH2M HILL Review Team Leader (RTL)
David Lane (CH2M HILL)

- o Sample Team Leader
Ned Pennock (CH2M HILL)
- o Laboratory Operation
Samples for RAS and SAS chemical analysis will be sent to an EPA CLP laboratory. Samples for conventional water analysis and selected metals will be sent to the ISBH laboratory.
- o Preparation of SAS requests
CH2M HILL
- o System/Performance Audits
 - CH2M HILL QA Manager (field)
 - U.S. EPA EMSL--Las Vegas (RAS and SAS--CLP)
 - U.S. EPA Quality Assurance Section (QAS) of the Monitoring and Quality Assurance Branch (MQAB) (ISBH)
- o Review of Tentatively Identified Compounds
Laboratory Scientific Support Section (LSSS), CRL
- o Final Review and Validation of CLP (RAS and SAS) Data, including Tentatively Identified Compounds
U.S. EPA Region V
Laboratory Scientific Support Section (LSSS)
Central Regional Laboratory (CRL)
- o CLP Data Completeness
CH2M HILL
- o Final Review and Validation of ISBH Data
CH2M HILL
- o ISBH Data Completeness
CH2M HILL
- o Final Review and Approval of QAPP
U.S. EPA, Region V QA officer

Primary responsibility for project quality rests with the SM. Independent QA review will be provided by the QA reviewers. A project organization chart is presented in Figure 4.

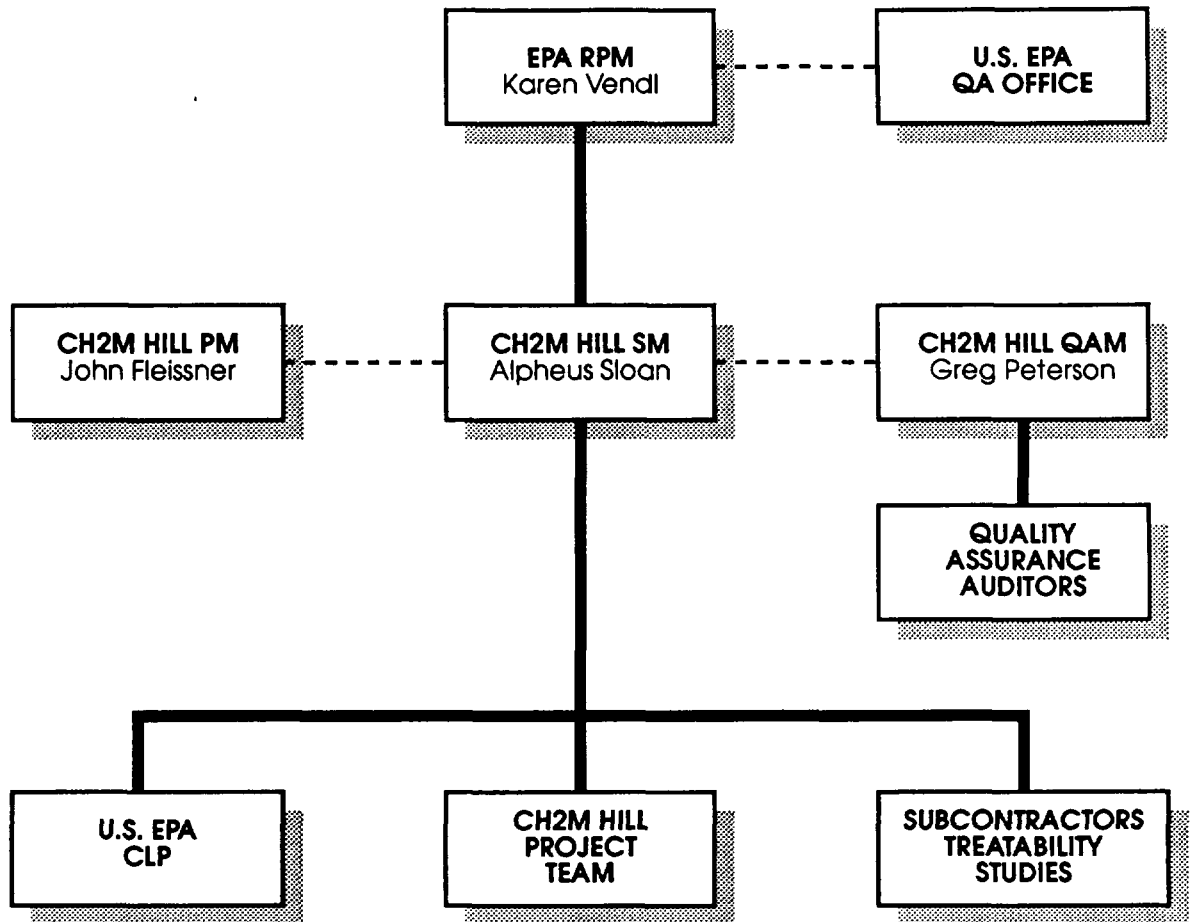


FIGURE 4
PREDESIGN PROJECT
ORGANIZATION
NSL/ECC QAPP

Section 3 QUALITY ASSURANCE OBJECTIVES

The overall QA objectives are to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide data for evaluating influent to be treated and to evaluate the effluent from the optimal treatment scheme proposed by the contractor. QA objectives for measurement data are to develop procedures for precision, accuracy, completeness, representativeness, and comparability. Specific procedures to be used for sampling, chain of custody, calibration, laboratory analysis, reporting, internal quality control, and its preventative maintenance and corrective actions are described in other sections of this QAPP.

FIELD QC AUDITS

Replicate and blank groundwater and leachate samples will be collected in the field and submitted to the CLP and ISBH laboratories to assess the quality of data from field sampling efforts. Replicate samples will be used to assess the combined effects of sample collection, handling, and analysis on data precision. Field blanks will be analyzed to check for procedural factors or ambient conditions at the site that may cause contamination. Trip blanks will be prepared for groundwater and leachate VOC samples to check for cross-contamination that may occur during sample storage or shipment. Numbers of both replicates and blanks are provided in Table 1, and methods of collection are specified in the Site Sampling Plan.

Replicate samples must be collected concurrently with actual samples in exactly equal volumes, at the same location, with the same sampling equipment, and in identical containers. They must also be preserved and handled in the same manner as the samples. Field blank samples will consist of deionized, contaminant-free water that is collected, containerized, treated, and handled in the same manner as the samples. The trip blank samples will be prepared offsite by filling clean VOA vials with ultrapure distilled water. They will accompany the groundwater and leachate samples to and through the CLP laboratory.

The number of replicate, field blank, and trip blank samples is listed in Table 1. Field blank samples and field replicate samples will be collected at the frequency of one per group of ten or fewer investigative samples. Trip blank

samples for groundwater and leachate VOCs will be collected at the frequency of one (two 40-ml vials) per each shipping cooler of water VOA samples.

Field measurements, including pH, specific conductivity, and temperature, involve data where QA concerns are appropriate but sample collection is not required. Procedures for conducting field measurements, calibrating equipment, and maintenance are documented in Appendix E. The primary QA objectives for field measurements are to obtain reproducible measurements with a degree of accuracy consistent with limitations of the analytical techniques used and with the intended use of the data.

ACCURACY, PRECISION, AND SENSITIVITY OF LABORATORY ANALYSIS

All groundwater and leachate samples will be analyzed by a CLP laboratory or the ISBH laboratory. The QA goals for RAS are established under CLP guidelines, as stated in the Statement of Work for Organics Analysis (July 1987) and the Statement of Work for Inorganics Analysis (July 1987). The quality control limits for accuracy and precision for CLP analyses are also stated in the Statements of Work. Besides routine organic and inorganic analyses, CLP Special Analytical Services (SAS) QA for groundwater and leachate samples are presented in Appendix C. QA goals for the ISBH laboratory are presented in Appendix D. QA goals for field measurements, including accuracy and precision, are documented in Appendix E.

COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

Data completeness can be quantified during data assessment. The laboratories are expected to provide data meeting QC acceptance criteria for 90 percent or more of the requested determinations.

The objective of representativeness is to assess whether the information obtained during the investigation accurately represents the actual site conditions. Requirements of representation were determined from previous investigations. Representativeness will be assessed after initial data validation.

The use of the standard sampling procedures and recognized field and laboratory techniques for sampling and analyses should make the resulting data comparable to other similar measurements on similar samples.

Date: 03/17/89
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METHOD DETECTION LIMITS

Contract required quantification limits for RAS are given in Appendix A, Table A-2. Contract required method detection limits for SAS are given in Appendix C. Detection limits for ISBH data are presented in Appendix D. The detection limits for field measurements are described in the procedures for operating the equipment (Appendix E).

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Section 4 SAMPLING PROCEDURES

Detailed sampling procedures are provided in the Site Sampling Plan (Appendix A). Table 1 contains a summary of sampling and analysis.

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Section 5 SAMPLE CUSTODY

INTRODUCTION

It is U.S. EPA and Region V policy to follow the U.S. EPA Region V sample custody or chain-of-custody protocols as described in "NEIC Policies and Procedures," EPA-330/9-78-001-R, revised June 1985. This custody is in three parts: sample collection, laboratory, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if the documents:

- o are in your possession
- o are in your view after being in your possession
- o were in your possession and you placed them in a secured location
- o are in a designated secure area

FIELD SPECIFIC CUSTODY PROCEDURES

The sample packaging and shipment procedures summarized below will ensure that the samples arrive at the laboratory with the chain-of-custody intact.

Field procedures are as follows:

1. The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.
2. All bottles will be tagged with sample numbers and locations. The Sample Management Office (SMO) number and stickers will be affixed.
3. Sample tags are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because a ballpoint pen would not function in freezing weather.

4. The contractor's site manager must review all field activities to determine whether proper custody procedures were followed during the field work and decide if additional samples are required. He or she should notify the U.S. EPA Remedial Project Manager of a breach or irregularity in chain-of-custody procedures.

Transfer of custody and shipment procedures are as follows:

1. Samples are accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.
2. Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and EPA custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.
3. Whenever samples are split with a source or government agency, a separate sample receipt is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "received by" space.
4. All shipments will be accompanied by the chain-of-custody record identifying the contents. The original record will accompany the shipment, and the pink and yellow copies will be retained by the sampler for return to the sampling office.
5. If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation.

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If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

LABORATORY CUSTODY PROCEDURES FOR THE CONTRACT LABORATORY

The RAS and SAS chain-of-custody procedures for the Contract Laboratory Program (CLP) are described in the appropriate SOWs.

FINAL EVIDENCE FILES CUSTODY PROCEDURES

The final evidence files from the CRL and Contract Laboratory Program are maintained by the Region V CRL Laboratory Support Team Data Coordinator.

The contractor maintains the files along with all relevant records, reports, logs, field notebooks with field measurements and HNu/OVA screenings, pictures, subcontractor reports, and LSSS data reviews in a secured, limited access area and under custody of the contractor's site manager.

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Section 6 ANALYTICAL SERVICES

All samples will be analyzed using RAS for TCL volatile organic compounds and inorganic chemicals, SAS for selected inorganic chemicals, and ISBH analyses for conventional parameters, arsenic and chromium (total and hexavalent). Field measurements will also be made. QAPP elements for each are listed below and documented in the references cited.

CLP ROUTINE ANALYTICAL SERVICES

SAMPLE CUSTODY

Sample custody procedures for CLP laboratory analysis are presented in Section 5.

ANALYTICAL AND CALIBRATION PROCEDURES

The CLP laboratory will analyze all samples collected for Target Compound List VOCs and metals. Testing of groundwater and leachate samples will conform to the guidelines in the User's Guide to the Contract Laboratory Program and to those specified in the statements of work for analysis of organic compounds and inorganic chemicals.

Calibration procedures for CLP RAS analysis are found in the Statement of Work for Organics Analysis (July 1987) and the Statement of Work for Inorganics Analysis (July 1987).

INTERNAL QUALITY CONTROL CHECK

Internal quality control procedures for groundwater and leachate samples will follow CLP guidelines specified in the statements of work for analysis of organic compounds and inorganic chemicals. Field blanks will be collected to check for sample contamination resulting from field sampling equipment, and replicates will be collected to check data precision.

DATA REDUCTION, VALIDATION, AND REPORTING

Data validation will be performed by the LSSS and the CRL QA Coordinator. The raw data collected from project sampling tasks and used in project reports will be appropriately identified and will be included in project files. Where test data have been reduced, the reduction method will be described. CH2M HILL will perform data reductions.

PERFORMANCE AND SYSTEMS AUDITS

RAS performance and systems audits are the responsibility of the U.S. EPA EMSL--Las Vegas.

The QAM will monitor and audit performance of the QA procedures to ensure that the project is performed in accordance with approved quality assurance procedures. The QAM will conduct the audits as described in Section 9, Audit Program, of the CH2M HILL REM/FIT Quality Assurance Manual. Audits may be scheduled at various times to evaluate the execution of sample identification, sample control, chain-of-custody procedures, field notebooks, and sampling procedures.

PREVENTATIVE MAINTENANCE

Preventative maintenance for laboratory instruments shall be performed as specified in the Statement of Work for Inorganics Analysis (July 1987) and the Statement of Work for Organics Analysis (July 1987).

DATA ASSESSMENT

Data assessment and data validation including TICs are the responsibility of the Laboratory Scientific Support Section (LSSS) of the U.S. EPA Region V Central Regional Laboratory (CRL). Data completeness will be checked by CH2M HILL and the SMO.

ACCURACY AND PRECISION DEFINITIONS

RAS accuracy and precision definitions for analyses performed are listed in the Statements of Work for analysis of organic compounds and inorganic chemicals.

CORRECTIVE ACTION

If QC audits find unacceptable conditions, the laboratory will contact the Program Coordinator of the LSSS. The remedial project manager and site project manager will be informed of the unacceptable conditions and, along with the LSSS, will develop and initiate the appropriate corrective action.

CLP SPECIAL ANALYTICAL SERVICES

CLP Special Analytical Services are being requested for chromium and copper analyses. Lower detection limits for chromium and copper are requested. These limits are based on the proposed State of Indiana NPDES permit for Northside

Sanitary Landfill (Appendix G) which requires that these discharge limits be met.

SAMPLE CUSTODY

Sample custody procedures for CLP laboratory analysis are presented in Section 5.

ANALYTICAL AND CALIBRATION PROCEDURES

The CLP laboratory will analyze all leachate and groundwater samples for selected metals (SAS). Testing will conform to the guidelines in the User's Guide to the Contract Laboratory Program and to the analytical and calibration procedures specified in the SAS requests (Appendix C).

INTERNAL QUALITY CONTROL CHECK

Internal quality control procedures for groundwater and leachate SAS sample analysis are listed in Appendix C. Field blanks will be collected to check for sample contamination resulting from field sampling equipment, and replicates will be collected to check data precision.

DATA REDUCTION, VALIDATION, AND REPORTING

Data validation will be performed by the LSSS and the CRL QA Coordinator. The raw data collected from project sampling tasks and used in project reports will be appropriately identified and will be included in project files. Where test data have been reduced, the reduction method will be described. CH2M HILL will perform data reductions.

PERFORMANCE AND SYSTEMS AUDITS

SAS performance and systems audits are the responsibility of the U.S. EPA EMSL--Las Vegas.

The QAM will monitor and audit performance of the QA procedures to ensure that the project is performed in accordance with approved quality assurance procedures. The QAM will conduct the audits as described in Section 9, Audit Program, of the CH2M HILL REM/FIT Quality Assurance Manual. Audits may be scheduled at various times to evaluate the execution of sample identification, sample control, chain-of-custody procedures, field notebooks, and sampling procedures.

PREVENTATIVE MAINTENANCE

Preventative maintenance for laboratory instruments shall be performed as specified in the Statement of Work for Inorganics Analysis (July 1987) and the Statement of Work for Organics Analysis (July 1987).

DATA ASSESSMENT

Data assessment and data validation including TICs are the responsibility of the Laboratory Scientific Support Section (LSSS) of the U.S. EPA Region V Central Regional Laboratory (CRL). Data completeness will be checked by CH2M HILL and the SMO.

ACCURACY AND PRECISION DEFINITIONS

SAS accuracy and precision requirements for analyses performed are listed in the SAS request forms (Appendix C).

CORRECTIVE ACTION

If QC audits find unacceptable conditions, the laboratory will contact the Program Coordinator of the LSSS. The remedial project manager and site project manager will be informed of the unacceptable conditions and, along with the LSSS, will develop and initiate the appropriate corrective action.

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SAMPLE CUSTODY

Sample custody includes three parts: sample collection, laboratory, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if the documents:

- o are in your possession
- o are in your view after being in your possession
- o were in your possession and you placed them in a secured location
- o are in a designated secure area

Field Specific Custody Procedures

The sample packaging and shipment procedures summarized below will ensure that the samples arrive at the laboratory with the chain-of-custody intact.

Field procedures are as follows:

1. The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.
2. All bottles will be labeled with sample number and location.
3. Sample labels are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because a ballpoint pen would not function in freezing weather.
4. The contractor's site manager must review all field activities to determine whether proper custody procedures were followed during the field work and decide if additional samples are required. He or she should notify the U.S. EPA Remedial Project Manager of a breach or irregularity in chain-of-custody procedures.

Transfer of custody and shipment procedures are as follows:

1. Samples are accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.
2. Samples will be properly packaged for shipment and dispatched to the laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with

clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

3. Whenever samples are split with a source or government agency, a separate sample receipt is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "received by" space.
4. If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

Laboratory Custody Procedures

Upon receipt of the samples in custody, the laboratory shall inspect the shipping container and sample bottles and shall document receiving information. The sample custodian or a designated representative shall sign and date all appropriate receiving documents at the time of receipt. The laboratory shall contact CH2M HILL if documents are absent, information on receiving documents does not agree, custody seals are not intact, or the sample is not in good condition. The contactor shall document resolution of any discrepancies, and this documentation shall become a part of the permanent case file.

Once samples have been accepted by the laboratory, they will be checked and logged in. They must be maintained in a secure sample storage or laboratory area.

Final Evidence Files Custody Procedures

The contractor maintains the files along with all relevant records, reports, logs, field notebooks with field measurements and HNu/OVA screenings, pictures, and subcontractor reports in a secured, limited access area and under custody of the contractor's site manager. ISBH will retain the evidence file containing the original laboratory analyses records.

ANALYTICAL AND CALIBRATION PROCEDURES

SAS Request Forms have been filled out with ISBH procedures attached for BOD₅, COD, TSS, VSS, TDS, nitrate, nitrite, TKN, ammonia, total phosphorus, alkalinity, chlorides, arsenic, chromium (total and hexavalent), and sulfates. This was done to ensure complete documentation of analysis and to document quality control procedures (Appendix D).

INTERNAL QUALITY CONTROL CHECK

Quality control requirements for each of the ISBH analyses are specified in Appendix D. Replicates and field blanks will be collected and submitted to ISBH for analysis. They will be used to check precision and to determine if any contamination is the result of field sampling procedures.

DATA REDUCTION, VALIDATION, AND REPORTING

The test procedures used are identified in Appendix D. Bench records and all records of analyses and calculations for samples, blanks, duplicates, spikes, and standards and resulting instrument readouts will be provided by the ISBH laboratory along with worksheets used to calculate results. The raw data collected and used in project reports will be appropriately identified and included in project files. CH2M HILL will perform data validation and reduction. Any method used for data reduction will be clearly described.

PERFORMANCE AND SYSTEMS AUDITS

Performance and systems audits of the ISBH laboratory are the responsibility of the U.S. EPA Quality Assurance Section (QAS) of the Monitoring and Quality Assurance Branch (MQAB). Systems audits and required performance limits are specified for each ISBH analysis in Appendix D.

The QAM will monitor and audit performance of the QA procedures to ensure that the project is performed in accordance with approved quality assurance procedures. The QAM will conduct the audits as described in Section 9, Audit Program, of the REM/FIT Quality Assurance Manual. Audits may be scheduled at various times to evaluate the execution of sample identification, sample control, chain-of-custody procedures, field notebooks, and sampling procedures.

DATA ASSESSMENT

Data assessment will be the responsibility of CH2M HILL.
Data completeness will be checked by CH2M HILL.

ACCURACY AND PRECISION DEFINITIONS

Accuracy and precision are specified for each ISBH analysis in Appendix D.

CORRECTIVE ACTION

If quality control audits detect unacceptable conditions or data, samples should be reanalyzed if holding time criteria permit. CH2M HILL should be contacted if requirements are not met upon reanalysis of samples.

FIELD ANALYSES

ANALYTICAL AND CALIBRATION PROCEDURES

Groundwater and leachate samples will be analyzed for pH, specific conductance, and temperature according to the procedures given in Appendix E. Analytical and calibration procedures for OVA/HNu screening for health and safety are found in Appendix E.

INTERNAL QUALITY CONTROL CHECK

Field analyses will be performed onsite and will not involve samples that are collected and retained. The primary QA/QC objective is to obtain reproducible measurements to a degree of accuracy consistent with limits imposed by analytical methodologies used and with the intended use of the data. Quality control procedures will be limited to checking the reproducibility of measurements by taking multiple readings and by calibration of instruments (where appropriate).

DATA REDUCTION, VALIDATION, AND REPORTING

All field recording sheets, instrument outputs, and worksheets for calculating results will be retained. Summarized raw data will be appropriately identified in reports.

PERFORMANCE AND SYSTEMS AUDITS

Instruments used to make field measurements will be calibrated as specified in Appendix E.

PREVENTATIVE MAINTENANCE

Preventative maintenance of field equipment is addressed in Appendix E.

DATA ASSESSMENT

The QAM will assess data to ensure that QA/QC objectives are met.

ACCURACY AND PRECISION DEFINITIONS

The system will be calibrated daily, and be demonstrated to have a precision of ± 30 percent.

CORRECTIVE ACTION

If variability among multiple readings at a single site is judged excessive, instruments will be recalibrated and the measurement repeated. If variability remains unacceptably high and instruments fail to calibrate properly, the QAM will be notified.

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Section 7
QUALITY ASSURANCE REPORTS TO MANAGEMENT

No separate QA report for this project is anticipated. The final report will contain separate QA sections that summarize data quality information collected during the project.

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Appendix A
SITE SAMPLING PLAN

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Appendix A SITE SAMPLING PLAN

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Appendix A
SITE SAMPLING PLAN

OBJECTIVE

This sampling plan documents procedures and practices to be followed during groundwater and leachate sampling for bench-scale treatability studies at the NSL/ECC site.

SAMPLE LOCATIONS, NUMBERS, AND ANALYSES

Samples for laboratory and bench-scale testing will be taken from leachate tank no. 2 on the site. Groundwater will also be sampled from the recently installed pump test well and ECCMW17. Five samples plus replicates and field blanks will be collected in the field at each sampling site, including the two wells and the leachate tank, and sent to a CLP laboratory for metal and VOC analyses and to the ISBH laboratory for conventional pollutant analysis and chromium and arsenic analysis. Ten groundwater samples (plus replicates and blanks) will be collected in the field and submitted for analyses. Five leachate samples (plus replicates and blanks) will be collected in the field and submitted for analyses. In addition, a large volume of water (approximately 55 gallons) will be collected at each location for bench-scale testing at a subcontracted laboratory.

Upon completion of the bench-scale studies, the final effluent from the contractor's optimized metals removal treatment process will be sent for CLP analysis for metals and VOCs and to the ISBH laboratory for conventional pollutant and arsenic and chromium analysis. Ten groundwater samples (plus replicates and blanks) from the optimized treatment process will be submitted for analyses. Five leachate samples (plus replicates and blanks) from the optimized treatment process will be submitted for analyses.

The number of samples and analyses are presented in Table 1 of the QAPP. A total of 20 groundwater samples and 10 leachate samples (plus replicates and blanks) will be sent for analyses. Sample containers and preservatives to be used for samples and blanks are specified in Table A-1. Sampling locations are shown in Figure 2 of the QAPP.

SAMPLE DESIGNATION

The sampling team leader will maintain a list of sample identification numbers in the sampling log book. A CH2M HILL

sample numbering system will be used to identify each sample for analysis, including duplicates and blanks. A Sample Management Office (SMO) number and a Central Region Lab (CRL) number will be assigned to each sample at the same time. Refer to the User's Guide to the Contract Laboratory Program for an explanation of the SMO numbers and to the CRL Sample Handling Manual for an explanation of the CRL numbers.

The designation NSL or ECC identifies the site where the samples will be taken. Each sample number will have a two-letter code corresponding to the sample type, followed by an alphanumeric or alpha sample location code. The letter codes are:

- o GW--groundwater well
- o LT--leachate tank

Field blanks will have "FB" following the letter code for the type of sample (e.g., a groundwater blank will be GWFB). Replicate samples will be identified by "R." Trip blanks for groundwater and leachate VOCs will have the designation "TB."

For example:

- o NSL-LT02: NSL site, from leachate tank No. 2
- o ECC-MW17: ECC site, from monitoring well 17

GENERAL SAMPLING AND TESTING PROCEDURES

GROUNDWATER SAMPLE COLLECTION

Before each well is purged for sampling, the water level will be measured using a stainless steel "popper." Each well to be sampled will be purged immediately before sampling using either a stainless steel or Teflon bailer, a submersible positive displacement pump, or a peristaltic pump. Well volumes will be calculated from the total depth of the well and the depth to water surface in the well. Discharge water will be collected and measured so that at least five well volumes are removed before sample collection. If pumps are used, the bottom 5 feet of hose will be made of Teflon so the hose will not contaminate the well or well water. All water purged from the well will be released onsite if the HNu or OVA readings are at background. If HNu or OVA readings are above background, water will be collected in 55-gallon drums for later disposal.

TABLE A-1
SAMPLE CONTAINERS AND PRESERVATIVES
NSL/ECC SITE

Sample Type	Analysis	Containers	Preservation	Holding Time	Quantity	Method of Shipment	Packing
Aqueous Low Level	RAS Organic Compounds						
	- Volatiles	Two 40-ml VOA vials	Iced to 4 C	7 days	Fill to top, no air space	Daily by overnight carrier	Vermiculite or poly-foam cooler
	RAS Inorganic Compounds						
	- Metals (including mercury)	One 1-liter polyethylene bottle	5ml/L HNO ₃ to pH < 2	6 months (26 days for mercury)	Fill to shoulder	Daily by overnight carrier	Vermiculite or poly-foam cooler
	SAS Inorganic Compounds						
	- Metals - Cr and Cu	One 1-liter polyethylene bottle	5ml/L HNO ₃ to pH < 2	6 months	Fill to shoulder	Daily by overnight carrier	Vermiculite or poly-foam cooler
	ISBH/SAS Conventional Parameters						
	- BOD	One 1-liter polyethylene bottle	Iced to 4 C	48 hours	Fill to shoulder	Daily by overnight carrier	Vermiculite or poly-foam cooler
	- COD, nitrate + nitrite, total Kjeldahl nitrogen, ammonia, total phosphorus	One 1-liter polyethylene bottle	2ml/L H ₂ SO ₄ to pH < 2 Iced to 4 C	28 days	Fill to shoulder	Daily by overnight carrier	Vermiculite or poly-foam cooler
	- Total suspended solids Volatile suspended solids Total dissolved solids Alkalinity Chlorides, sulfates	One 1-liter polyethylene bottle	Iced to 4 C	7 days 7 days 7 days 14 days 28 days	Fill to shoulder	Daily by overnight carrier	Vermiculite or poly-foam cooler
Aqueous Medium Level	ISBH/SAS Metals						
	- Chromium, hexavalent	One 500 ml polyethylene bottle	Iced to 4 C	24 hours	Fill to shoulder	Daily by overnight carrier	Vermiculite or poly-foam cooler
	- Chromium, total and arsenic	One 500 ml polyethylene bottle	5ml/L HNO ₃ to pH < 2	6 months			
	RAS Organic Chemicals						
	- Volatiles	Two 40-ml VOA vials	Iced to 4 C	7 days	Fill to top, no air space	Federal Express Priority 1 with restricted article paperwork	In cans with vermiculite

Note: Aqueous samples for matrix spike and matrix spike duplicates will require additional volume of samples and bottles for analyses. For VOC analysis, triple the normal volume of samples will be collected.

After the well has been purged, the samples for VOCs will be collected using a dedicated bottom loading stainless steel or Teflon bailer. The bailers will be raised and lowered on a thin stainless steel cable. Samples for metals and conventional pollutant analysis will be collected using a bailer or a pump.

LEACHATE SAMPLE COLLECTION

The tank will be sampled using a diaphragm pump. The discharge tubing in the tank will be weighted at the end to keep it submerged and minimize the transfer of VOCs through open air discharge. The pump will be equipped with tygon flexible tubing for collection of samples for metal and conventional pollutant analysis. VOC samples will be drawn from the observation/pumpout port of the tank using a dedicated bailer.

REPLICATE SAMPLE, FIELD BLANK SAMPLE, TRIP BLANK SAMPLE, AND MATRIX SPIKE SAMPLE PREPARATION

Replicate, field blank, and matrix spike samples will be collected in the field and submitted to the CLP and ISBH laboratories to assess the quality of the data. Replicate samples will be used to assess the combined effects of sample collection and handling and for analysis of data precision. Field blanks will be analyzed to determine the extent to which field procedures contribute to sample contamination. Trip blanks will be prepared for groundwater and leachate VOCs only to determine if cross-contamination occurred during sample storage and shipment.

Replicate samples must be collected at the same time, at exactly the same location, with the same apparatus, and into identical containers prepared in the same way and filled to the same volume. All replicate samples must be preserved and handled identically. Field blank samples are deionized, contaminant-free water that is collected, containerized, treated, and handled in the same manner as the samples. Trip blanks will be prepared by transferring deionized water from the ISBH laboratory into the VOA vials. Matrix spike samples are additional samples to be spiked with certain chemicals in the CLP and ISBH laboratories to ensure the quality of the analytical process.

The sample replicates, field blanks, trip blanks, and matrix spikes are listed in Table 1 of the QAPP. The number of matrix spike samples and trip blanks is not included in the total number of samples. For groundwater and leachate, one

replicate sample and one field blank sample will be collected at a frequency of one per group of ten or fewer investigative samples. For trip blanks, one blank will be prepared for each shipping cooler of water VOC samples.

Field and trip blanks will be prepared using reagent grade deionized water from the ISBH laboratory. Groundwater field blanks will be prepared by pouring deionized water into a decontaminated bailer and then transferring it to appropriate sample containers. The metal field blank will be unfiltered. Trip blanks will be prepared by filling VOA vials with deionized water, capping them in the laboratory, and shipping them with sample containers to the field. Trip blanks will remain capped during sampling. When sampling is done, the trip blanks will be shipped with VOA samples. All field blanks will be preserved and handled as if they were actual samples.

EQUIPMENT DECONTAMINATION PROCEDURES

All sampling and well purging equipment (pumps and bailers) will be dedicated to that specific well or leachate tank. Following sample collection, decontamination will consist of washing the equipment in a solution containing 2.5 percent (w/w) trisodium phosphate followed by a 10 percent (v/v) solution of methanol and deionized water, and finally a triple rinse with deionized water. When dedicated pumps are used, the decontamination solutions will be pumped through the pump and hoses. Sampling equipment will be placed on clean plastic sheeting and allowed to air dry before being used again.

SAMPLE HANDLING AND ANALYSES

PARAMETERS

The parameters of sample analysis are listed in Table 1 and include RAS TCL volatile organic compounds and TCL metal analysis. Special analytical services were requested for chromium and copper as lower detection limits are required. Conventional pollutants and arsenic and chromium will be analyzed by the ISBH laboratories. A list of TCL volatile organic compounds and metals which will be analyzed are presented in Table A-2. Samples for VOA analysis may be low or medium concentration samples as determined in the field. The SMO will be notified as soon as the determination is made so that the designated CLP laboratory can be notified.

Temperature, pH and specific conductance will be measured in the field for all groundwater and leachate samples. The

Table A-2
 TARGET COMPOUND LIST AND
 CONTRACT REQUIRED QUANTIFICATION LIMITS (Page 1 of 2)

Volatiles	CAS Number	Quantification Limits Low Concentration Analysis ^a	
		Water (ug/l)	Soil/Sediment ^b (ug/kg)
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-34-4	5	5
9. 1,1-Dichloroethane	75-35-3	5	5
10. 1,2-Dichloroethene (total)	540-59-0	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-2	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	75-27-4	5	5
18. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
19. 1,2-Dichloropropane	78-87-5	5	5
20. Cis-1,3-Dichloropropene	10061-02-5	5	5
21. Trichloroethene	79-01-6	5	5
22. Dibromochloromethane	124-48-1	5	5
23. 1,1,2-Trichloroethane	79-00-5	5	5
24. Benzene	71-43-2	5	5
25. Trans-1,3-Dichloropropene	10061-01-6	5	5
26. Bromoform	75-25-2	5	5
27. 2-Hexanone	591-78-6	10	10
28. 4-Methyl-2-pentanone	108-10-1	10	10
29. Tetrachloroethene	127-18-4	5	5
30. Toluene	108-88-3	5	5
31. Chlorobenzene	108-90-7	5	5
32. Ethylbenzene	100-41-4	5	5
33. Styrene	100-42-5	5	5
34. Xylenes (total)	133-02-7	5	5

Note: Specific quantification limits are highly matrix dependent. The quantification limits listed herein are provided for guidance and may not always be achievable.

^aQuantification limits listed for soil/sediment are based on wet weight. The quantification limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

^bMedium Soil/Sediment Contract Required Quantification Limits (CRQL) for Volatile TCL Compounds are 100 times the individual Low Soil/Sediment CRQL; for Semivolatile TCL Compounds they are 60 times the individual Low Soil/Sediment CRQL.

Inorganic Target Analyte	Quantification Limit
	Low Concentration Analysis ^a (ug/l)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5,000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	5
Magnesium	5,000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5,000
Selenium	5
Silver	10
Sodium	5,000
Thallium	10
Vanadium	50
Zinc	20
Cyanide	10

^aThe quantification limits for samples may be considerably higher depending on the sample matrix.

procedures of field measurement are described in Appendix E, including OVA and HNu monitoring for health and safety.

SAMPLE PREPARATION

Preparation procedures for samples are summarized in Table A-1. All preservatives will be prepared using reagent grade chemicals. Samples for chemical analysis will be kept iced to about 4°C to preserve the samples as indicated on Table A-1.

SAMPLING TEAM MEMBERS AND SCHEDULE

The following responsibilities have been assigned for the field sampling activities:

- o Site Manager (SM)
Alpheus Sloan/CH2M HILL
- o Sampling Team Leader
Ned Pennock/CH2M HILL

The other sampling team members will be from CH2M HILL.

The fieldwork schedule has not yet been determined, but it will last approximately 1 week. The LSSS/CRL will be notified approximately 1 month before sampling begins.

SAMPLING DOCUMENTATION

All samples will be collected under chain-of-custody procedures. Standard paperwork, including sample tags, traffic reports, chain-of-custody forms, and custody seals used for CLP sample tracking and records, will be filled out as described in Appendix F. Information about the samples will be entered in the site log maintained by the Sampling Team Leader. That information will include sample time, location, tag numbers, designation, and sampler. New readings, weather conditions, and field modifications or decisions will also be recorded. Entries will be made in ink unless weather conditions dictate otherwise. Photographs taken, along with the time, data, location, and task description, will also be noted in the log book.

WASTE DISPOSAL

Wastes generated during sampling will consist of well purge water, wastes from decontamination, and protective clothing. Wastes with HNu and OVA readings above background will be collected in 55-gallon drums approved by the Department of

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Transportation. The drums will be labeled and stored temporarily stored onsite for later disposal.

GLT821/11

Appendix B
EXISTING DATA

GLT821/15-1

Table 7
GROUNDWATER INORGANIC ANALYTICAL RESULTS - PHASE I
ECC SITE (SUBTASK 3-2)
CASE NO. 1838

Compound ^a	MS0283 1A-001	MS027 1C-001	MS0284 2A-001	MS0271 2B-001	MS0272 2C-001	MS0285 3A-001	MS0288 3A-002	MS0273 3C-001	MS0274 4C-001	MS0275 4C-002	MS0286 5A-001	MS0276 BD-001
Aluminum ^b	ND	ND	ND	ND	ND	830	320	ND	ND	ND	1,720	ND
Chromium	ND	ND	ND	ND	ND	13	ND	ND	ND	ND	11	ND
Barium ^b	306	660	330	150	380	570	560	210	510	510	390	ND
Beryllium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Cobalt	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Copper	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Iron	2,390	600	2,740	920	670	8,300	6,330	1,820	850	970	7,410	ND
Nickel	ND	ND	ND	ND	ND	42	77	ND	42	52	ND	ND
Manganese	110	22	56	54	17	260	230	51	ND	22	161	ND
Zinc	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Boron ^c	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Vanadium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Silver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Arsenic	ND	ND	ND	ND	ND	19	20	ND	ND	ND	ND	ND
Antimony	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	4	ND
Selenium	ND	ND	ND	ND	ND	3	4	ND	ND	ND	ND	ND
Thallium ^b	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.4	0.4 ^c
Mercury	0.4 ^d	ND	0.3 ^d	0.3 ^d	ND	0.3 ^d	ND	ND	ND	ND	ND	11.2 ^c
Tin ^{b,c}	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Cadmium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Lead	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Cyanide	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

^aAll concentrations expressed in ug/L.

^bQA data indicate poor or marginal recovery of these spiked metals.

^cQA data indicate the presence of these metal contaminants in the laboratory method blank.

^dThis metal was also detected in the analysis of the field blank.

ND = Not detected.

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Table 8
GROUNDWATER INORGANIC ANALYTICAL RESULTS - PHASE II
ECC SITE (SUBTASK 3-2)
Case No. 2197

Compound ^a	MS0927 1A-01	MS0298 1A-02	MS0929 1C-01	MS0930 2A-01	MS0921 2B-01	MS0932 2C-01	MS0933 3A-01	MS0934 3C-01	MS0935 4C-01	MS0936 5A-01	MS0937 6A-01	MS0938 7A-01	MS0939 7A-02	MS0940 99-01
Aluminum	<200	406	<200	<200	<200	<200	<200	<200	<200	361	<200	61,500	663	<200
Chromium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	144	ND	ND
Barium	366	357	657	268	188	470	1,070	264	563	392	508	875	397	<100
Beryllium	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
Cobalt	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	80	<50	<50
Copper	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	106	<50	<50
Iron	3,070	3,300	736	3,360	1,140	874	10,400	1,720	108	328	5,470	105,000	1,030	210
Nickel ^b	<40	<40	<40	<40	<40	<40	80	<40	<40	<40	<40	176	<40	<40
Manganese	<103	95 ^c	28	49	54	23	97	39	23	52	231	1,930	113	<10
Zinc	45	14 ^c	19	11	ND	26	19	ND	74	36	35	276	31	49
Boron	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Vanadium	<200	<200 ^c	<200	<200	<200	<200	<200	<200	<200	<200	<200	<200	<200	<200
Silver	25	14 ^c	<10	<10	27	33	<10	25	19	<10	<10	<10	<10	20
Arsenic	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Antimony	<20	<20 ^d	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Selenium	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2
Thallium	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Mercury	<0.2	<0.2 ^d	<0.2	<0.2	<0.2	0.4	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	0.8
Tin	<20	<20 ^d	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Cadmium	<1	<1 ^d	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Lead	<5	<5 ^d	<5	<5	<5	<5	<5	<5	<5	<5	<5	102	<5	<5
Cyanide	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10

^aAll concentrations expressed in ug/l.

NA = Not analyzed for.

ND = Not detected.

^bQA data indicate that these metals may be high by 25 to 30% based on ICAP intercheck.

^cQA data indicate that relative percent differences (RPD's) are outside accepted QA limits for these metals.

^dQA data indicate that matrix spike recoveries for these analyses are below accepted QA limits.

Table 9
GROUNDWATER INORGANIC RESULTS - PHASE III
ECC SITE (SUBTASK 3-2)
Case No. 3606

Compound ^a	ME4629 1A-001	ME4628 2A-001	ME4625 3A-001	ME4622 5A-001	ME4630 5A-002	ME4627 6A-001	ME4626 7A-001	ME4631 8A-001	ME4632 BLANK	ME4624 10A-001
Aluminum	304	[65]	[128]	ND	[140]	[66]	[77]	[144]	[57]	[72]
Antimony	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Arsenic	ND	ND	15	ND	ND	ND	ND	ND	ND	ND
Barium	328	287	868	413	438	612	331	353	ND	298
Beryllium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Cadmium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Calcium	95770 E	98200 E	70240 E	94890	99410 E	161100 E	73550 E	98500 E	[900] E	77000 E
Chromium	11	11	15	13	12	ND	ND	ND	ND	ND
Cobalt	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Copper	ND	ND	[16]	ND	ND	ND	ND	ND	ND	ND
Iron	1454	2931	297	202	356	1194	[73]	2545	[98]	[51]
Lead	6.7	ND	ND	ND	ND	ND	6.5	ND	ND	ND
Cyanide	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Magnesium	34660 E	32070 E	131800 E	33140 E	34160 E	69730 E	29780 E	38890 E	[334] E	31440 E
Manganese	66	49	70	73	50	94	57	24	ND	40
Mercury	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Nickel	ND	65	84	[32]	ND	46	ND	ND	[34]	ND
Potassium	ND	ND	105940	ND	ND	[2129]	[2625]	[1195]	ND	[4765]
Selenium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Silver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Sodium	10060	15490	380700	10980	11210	118000	22300	15130	1424	25520
Thallium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Vanadium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Zinc	69	260	250	155	158	42	37	69	31	54

FOOTNOTES:

- E - Value is estimated or not reported due to the presence of interference.
- R - Soike sample recovery is not within control limits.
- * - Duplicate analysis is not within control limits.
- + - Correlation coefficient for method of standard addition is less than 0.995.
- [] - Positive values less than the contract required detection limit.
- ND - Not detected

Table 10
GROUNDWATER ORGANIC ANALYTICAL RESULTS - PHASE I
ECC SITE (SUBTASK 3-2)
CASE NO. 1838

Compound ^a	S2383 1A-001	S2370 1C-001	S2384 2A-001	S2371 2B-001	S2372 2C-001	S2388 3A-001	S2373 3A-002	S2374 3C-001	S2375 4C-001	S2375 4C-002	S2386 5A-001	S2346 BK-001
BASE/NEUTRAL COMPOUNDS ^b												
fluoranthene							<20					
isophorone						<20	<20					
chrysene							<20					
pyrene							30					
diethylphthalate							<20					
VOLATILES ^c												
1,1,1-trichloroethane						<5						
1,1-dichloroethane						96	86					
chloroethane						120	116					
trans-1,2-dichloroethene						19	16					
methylene chloride						8		<5				34
trichloroethene						9	7					
vinyl chloride						7	6					
NONPRIORITY POLLUTANTS												
HAZARDOUS SUBSTANCES												
acetone			640		<100	1,400	1,400				490	
styrene					<5							
o-xylene			9									

^aExpressed as ug/l

^bQA data indicate the average surrogate recovery is <40% and these results are semiquantitative.

^cQA data indicate that these analyses were run after expiration of the acceptable holding period, however they are considered acceptable due to good analytical QA results.

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Table 11
GROUNDWATER ORGANIC ANALYTICAL RESULTS - PHASE II
ECC SITE (SUBTASK 3-2)
CASE NO. 2197

Compound ^a	S2803 1A-01	S2801 1A-02	S2802 1C-01	S2804 2A-01	S2805 2B-01	S2806 2C-01	S2807 3A-01	S2808 3C-01	S2809 4C-01	S2810 5A-01	S2811 6A-01	S2812 7A-01	S2813 7A-02	S2814 Blank
BASE/NEUTRAL COMPOUNDS														
bis(3-ethylhexyl)phthalate		<23												
VOLATILES														
benzene							<9							
1,1-dichloroethane							51.2							
chloroethane							40.7							
chloroform							<9							
trans-1,2-dichloroethene							<9							
methylene chloride	<9	<9	<9	11.0	<9	<9	18	12.4	<9	9.0	19.5	16.5	9.0	9.0 ^b
fluorotrichloromethane														11.6
tetrachloroethene	<9	<9		<9						9.0				
toluene														<9
trichloroethene	<9	<9					<9			<9				
vinyl chloride							85.8							
NONPRIORITY POLLUTANTS														
HAZARDOUS SUBSTANCES														
acetone	9,749	9,897	<9	3,016	<9	<9	15,030	550.7	<9	54.5	4,284	23.9	38.3	<9 ^b
2-butanone	<9													
o-xylene							12					<9		

^aExpressed as ug/l

^bQA data indicate that these compounds may have originated as laboratory contaminants.

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GLT360/11-1

Table 12
GROUNDWATER ORGANIC RESULTS - PHASE III (ug/L)
ECC SITE (SUBTASK 3-2)
CASE NO. 3606

Compound	E7485 11A-001	E7486 5A-001	E7487 9A-001	E7488 10A-001	E7489 3A-001	E7490 7A-001	E7491 6A-001	E7492 2A-001	E7493 1A-001	E7494 5A-001	E7495 8A-001	E7496 BLANK
BASE/NEUTRAL COMPOUNDS												
Bis (2-ethylhexyl)phthalate												59
VOLATILES												
benzene					4 J	4 J						
1,1,1-trichloroethane											7	
chloroethane				29		90						
chloroform							3 JB					6
1,1-dichloroethane				8	10	9					6	
trans-1,2-dichloroethane	4000			3 J	3 J						13	
ethylbenzene					3 J	4 J						
methylene chloride		3 J	2 J	4 J	7	3 J		3 J	22	4 J	64	
chloromethane					100							
trichloroethene	28000		3 J								21	
acetone			41B	53B			24B				52B	180
2-butanone				26B								10

FOOTNOTES:

- B Analyte has been found in the laboratory blank as well as the sample. Indicates probable contamination.
J Indicates an estimated value. When the mass spectral data indicates the presence of a compound that meets the identification criteria and the result is less than the specified detection limit but greater than zero.
Blanks indicate not detected.

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GLACIAL TILL WATER BEARING ZONE

Sample Locations	EA085-01	EA095-01	EA105-01	EA115-01	EA144-01	EA151-01	EA161-01	EA181-01	EA22-01	EA23-01	EA24-01
Sample Types									FIELD BLANK	FIELD BLANK	FIELD BLANK
Date Sampled	4-15-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-21-85	4-15-85
OIR Number	ED167	EA344	EA346	EA340	EA352	EA353	EA354	EA355	EA361	EA359	ED169
IIR Number	ME0357	ME0352	ME0354	ME0356	ME0340	ME0351	ME0342	ME0343	ME0349	ME0350	ME0359

ORGANIC COMPOUNDS (ug/l)

VOLAIILES

[illegible]

BASE/NEUTRALS and ACIDS

2-METHYLNAPHTHALENE	0.1	J		10	J		65	110	10	J				
2-METHYLPHENOL				10	J									
4-METHYLPHENOL				10	J									
BIS(2-ETHYLHEXYL)PHOSPHATE						10	J	10	J	10	J			
ETHYL BENZYL PHOSPHATE														
<hr/>														
DI-N-BUTYL PHOSPHATE				10	J, D	10	J, D	10	J, D	10	J, D	10	J, D	
DIETHYL PHOSPHATE														
NAPHTHALENE				10	J		33	28	10	J				
PHENOL				10	J									
PHENANTHRENE								10	J					
<hr/>														
TOTAL BASE/NEUTRALS and ACIDS	0.1	0	60	20	20	110	172	40	10			0		
<hr/>														
TOTAL TENTATIVELY IDENTIFIED BASE/NEUTRALS and ACIDS	276	J	70.3	J	137.0	J	972	J	3195	J	709	J	26	J

PESTICIDES and PCBs

[illegible]

TABLE B
NORTHSIDE LANDFILL GROUNDWATER DATA
PHASE I SAMPLING
REMEDIATION INVESTIGATION REPORT

GLACIAL TILL WATER BEARING ZONE

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Sample Location: Sample Type: Date Sampled: OIR Number: IIR Number:	GM08S-01 4-15-85 ED167 ME0357	GM09S-01 2-20-85 EA344 ME0532	GM10S-01 2-20-85 EA346 ME0534	GM119-01 2-20-85 EA348 ME0536	GM114-01 2-20-85 EA352 ME0540	GM115-01 2-20-85 EA353 ME0541	GM116-01 2-20-85 EA354 ME0542	GM118-01 2-20-85 EA355 ME0543	GM22-01 FIELD BLANK 2-20-85 EA361 ME0549	GM23-01 FIELD BLANK 2-21-85 ME0550	GM24-01 FIELD BLANK 4-15-85 ED169 ME0359
INORGANIC COMPOUNDS (ug/l)											
ALUMINUM	(63)			(24)	(32)			(33)			
ANTIMONY					21						
ARSENIC					1110						
BARIUM	(169)	362	(111)	(84)		(69)	(94)	(176)		(1.2)	
BERYLLIUM								(1.2)	(1.3)		
CALCIUM	31000	167000	220000	88600	182000	77300	91300	181000	(92)	(24)	
CHROMIUM		(3.7)									
COBALT		(9.4)	(7.7)	(6.1)	(9.8)			(17)			
COPPER		(8.3)						(9.4)			
IRON	(79)	(74)	(18)	(60)	17000	(23)	(8.2)	797	(30)	(6.6)	
LEAD	9.4			30		28	33	(3.9)			
CYANIDE		(2.5)	NR								
MANGANESE	27700	90000	67100	26600	165000	22900	30500	73700	(36)	(6.5)	
MANGANESE	140	490	912	112	851	204	185	4330			(3.2)
NICKEL		87	(32)		53			(35)			
POTASSIUM	5400	13500	10000	(4900)	152000	(1450)	(2620)				
SILVER									(4.9)		
SODIUM	26600	335000	142000	30100	531000	8930	14200	38000	(80)	(125)	(1550)
TIN											
VANADIUM					(3.6)	1		(4.0)			
ZINC	(19)	(8.8)	290	(16)	(3.7)	(17)		(16)	(5.6)		(7.7)
pH											
PERCENT MOISTURE (%)											
PERCENT SOLIDS (%)	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
OIL AND GREASE (ug/l)	40	16.3		3.15	182	6.46	49.5	11.5			
TOTAL ALKALINITY (ug/l as CaCO3)	5750	1406		907	1939.7	607	901.5	882.4			
CHLORIDE (ug/l)	5	625		23	964	23.6	21.6	100			
TOTAL PHOSPHORUS (ug/l as P)											
DISSOLVED SOLIDS (ug/l)	330	1751		445	2925	202	339	823		63	20
SULFATE (ug/l)		54.5		96.7	10.5	19.1	19.1	172			

FOOTNOTES:

- B: Analyte has been found in the laboratory blank as well as the sample. Indicates possible/probable contamination.
- C: Applies to pesticide parameters where the identification has been confirmed by GC/MS.
- E: Value is estimated due to presence of interference.
- J: An estimated value.
- K: Actual value, within the limitations of the method, is less than the given value.
- R: Spike sample recovery is not within control limits.
- S: Value is determined by standard addition.
- u: Duplicate analysis is not within control limits.
- uu: Sample(s) analyzed at medium concentrations.
- v: Correlation coefficient for method of standard addition is less than 0.995.
- l: Positive values less than the contract required detection limit.
- NR: Not required by contract at this time.

TABLE 8
NORTHSIDE LANDFILL GROUNDWATER DATA
PHASE I SAMPLING
REMEDIATION INVESTIGATION REPORT

SAND AND GRAVEL WATER BEARING ZONE

PAGE 2 OF 3

Sample Location:	GW001-01	GW019-01 GW001-01-DUP	GW002-01	GW003-01	GW004-01	GW005-01	GW006-01	GW020-01 GW006-01-DUP	GW007-01	GW021-01 GW007-01-DUP
Sample Type:										
Date Sampled:	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85
OTA Number:	E9262	EA358	E9263	E9264	E9265	EA339	EA340	EA359	EA341	EA360
TIN Number:	ME1864	MECS46	ME1865	MECS25	MECS26	MECS27	MECS28	MECS47	MECS29	MECS48
.....										
INORGANIC COMPOUNDS (ug/l)										
.....										
ALUMINUM										
ANTIMONY		(35)								
ARSENIC	15	15								
BARIUM	1490	1460	243	211	(152.1)	(171)	311	299	229	240
BERYLLIUM										
CALCIUM	89300	86000	53800	43700	25000	90600	63100	64400	79000	80400
CHROMIUM		(4.4)				(3.9)	(3.0)			
COBALT	(0.7)	(11)								
COPPER					(2.9)	(3.0)	(2.6)		(2.6)	
IRON	7530	6730	363	(111)	(38)	(71)	406	446	262	276
LEAD										
CYANIDE										
MAGNESIUM	114000	110000	20600	23900	12400	26000	30700	28900	28500	29200
MANGANESE	150	147	50	81	(12)	36	37	37	72	67
NICKEL	87	80								
POTASSIUM	108800	107000		(1480)	(1040)	(717)	(1670)	(592)	(1000)	
SILVER			(3.1)					(3.0)	(4.0)	
SODIUM	412000	410000	37200	39800	82200	6540	21700	28000	14300	14800
TIN									(15)	
VANADIUM										
ZINC	41	42	(17)	(5.7)	(5.9)		(3.4)	(3.2)	(11)	
.....										
pH										
PERCENT MOISTURE (%)										
PERCENT SOLIDS (%)	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
OIL AND GREASE (mg/l)	0.01	1.01		20.6	26	26.7	24	0.45	3.7	0.43
TOTAL ALKALINITY (mg/l as CaCO3)	917.6	950.7	293.5	274.4	303.5	305.5	301.9	371.9	335.7	344.7
CHLORIDE (mg/l)	781	821	5.7	6.3	7	14	1.7	1.7	10.6	10.7
TOTAL PHOSPHORUS (mg/l as P)										
DISSOLVED SOLIDS (mg/l)	2137	2104	239	376	360	313	246	204	283	325
SULFATE (mg/l)	15	13.0	1		1	96.7	1	1.4	20.7	22.3

FOOTNOTES:

- B: Analyte has been found in the laboratory blank as well as the sample. Indicates possible/probable contamination.
- C: Applies to pesticide parameters where the identification has been confirmed by GC/MS.
- E: Value is estimated due to presence of interference.
- J: An estimated value.
- K: Actual value, within the limitations of the method, is less than the given value.
- R: Spike sample recovery is not within control limits.
- S: Value is determined by standard addition.
- si: Duplicate analysis is not within control limits.
- si: Sample(s) analyzed at medium concentrations.
- si: Correlation coefficient for method of standard addition is less than 0.995.
- []: Positive values less than the contract required detection limit.
- NR: Not required by contract at this time.

END AND GRUEL WATER REMAINS DUE

Sample Location

SAMPLE 1 PER
Date Sampled
Old Number

Ulrich Hubert
Jill Hubert

ORGANIC COMPOUNDS (by/1)

VOLAILLES

[illegible]

TABLE B
NORTHSIDE LANDFILL GROUNDWATER DATA
PHASE I SAMPLING
REMEDIATION INVESTIGATION REPORT

SAND AND GRAVEL WATER BEARING ZONE

PAGE 3 OF 3 (cont.)

Sample Location:	GM00D-01	GM09D-01	GM10D-01	GM11D-01	GM12D-01	GM13D-01	GM06S-01	GM22D-01 FIELD BLANK	GM23D-01 FIELD BLANK	GM24D-01 FIELD BLANK
Sample Type:										
Date Sampled:	4-15-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-21-85	4-15-85
QIR Number:	ED168	EA345	EA347	EA349	EA350	EA351	EA356	EA361		ED163
IR Number:	ME0358	ME0353	ME0355	ME0357	ME0358	ME0359	ME0354	ME0359	ME0350	ME0359
.....										
INORGANIC COMPOUNDS (ug/l)										
.....										
ALUMINUM	(50)	(77)					(98)			
ANTIMONY		(30)					(50)			
ARSENIC	37	12					20			
BARIUM	1961	440	(157)	268	830	576	1580		(1.2)	
BERYLLIUM							(1.1)	(1.3)		
CALCIUM	35100	106000	31700	40000	92200	131000	192000	(92)	(24)	
CHROMIUM		(4.9)					(9.9)			
COBALT					(6.7)		(9.0)			
COPPER	(5)		(39)	(37)	184	1670	10400	(9.4)	(6.6)	
IRON	(71)	(77)					(30)			
LEAD	10									
CYANIDE		(3.6)					(5.3)			
MAGNESIUM	19500	41000	14900	23200	157000	89500	169000	(36)	(6.5)	
MANGANESE	93	125	61	36	340	1300	85			(3.2)
NICKEL		(24)			84	50	47			
POTASSIUM	(1400)	7400	(3420)	(1750)	55500	27700	147000			
SILVER							(5.6)			
SODIUM	32800	119000	69300	39000	426000	250000	536000	(1.9)	(125)	(1550)
TIN				(13)				(80)		
Vanadium		(3.3)					(10)			
ZINC	(13)			25		37	21	(5.6)		
.....										
Wt										
PERCENT MOISTURE (%)	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
PERCENT SOLIDS (%)										
OIL AND GREASE (ug/l)	20	4.7	3.7	4.35	8.52	1.23	8.75			
TOTAL ALKALINITY (ug/l as CaCO3)	49	2000	348.7	357.8	1050.2	762.8	1155.8			
CHLORIDE (ug/l)	7	242	9	2.3	1020	505	1070			
TOTAL PHOSPHORUS (ug/l as P)										
DISSOLVED SOLIDS (ug/l)	320	749	247	227	2096	1412	3331		63	20
SULFATE (ug/l)		11.6	4.5		14	88.8	6.5			

FOOTNOTES:

- D: Analyte has been found in the laboratory blank as well as the sample. Indicates possible/probable contamination.
- C: Applies to pesticide parameters where the identification has been confirmed by GC/MS.
- E: Value is estimated due to presence of interference.
- J: An estimated value.
- K: Actual value, within the limitations of the method, is less than the given value.
- R: Spike sample recovery is not within control limits.
- S: Value is determined by standard addition.
- ai: Duplicate analysis is not within control limits.
- aii: Sample(s) analyzed at medium concentrations.
- aii: Correlation coefficient for method of standard addition is less than 0.995.
- (j): Positive values less than the contract required detection limit.
- NR: Not required by contract at this time.

NORTHSHORE SURFACIAL TAIL WATER BEARING UNIT
PHASE 1 SAMPLING
REMEDIATION INVESTIGATION REPORT

PAGE 2 OF 4

Sample Location:	NSL 85A	NSL 95	NSL 105	NSL 115	NSL 14	NSL 15	NSL 16	NSL 18
Sample Number:	GM085-01	GM095-01	GM105-01	GM115-01	GM014-01	GM015-01	GM016-01	GM018-01
Date Sampled:	4-15-05	2-20-05	2-20-05	2-20-05	2-20-05	2-20-05	2-20-05	2-20-05
OTR Number:	ED167	ER344	ER346	ER352	ER353	ER354	ER355	ER356
IR Number:	ME0357	ME0332	ME0334	ME0336	ME0340	ME0341	ME0342	ME0343
INORGANIC COMPOUNDS (ug/l)								
ALUMINUM	(63)		(24)					
ANTIMONY								
ARSENIC				(32)	2		(33)	
BARIUM				110		(69)		
BERYLLIUM	(169)	362	(111)	(64)				
BROMINE								
CADMIUM								
CHROMIUM	54000	167000	220000	80500	182000	77300	91300	101000
COPPER		(19.4)	(7.7)	(6.1)	(19.0)		(117)	(9.4)
IRON	(79)	(74)	(10)	(60)	17000	(23)	(0.2)	797
LEAD	9.4	(2.5)	NR	30	20	33		
MANGANESE	27700	90000	67400	26600	165000	22900	30500	73700
NICKEL	140	490	912	112	55	204	105	4330
POTASSIUM	5400	13500	10000	(1900)	152000	(1450)	14200	30000
SILVER								
SODIUM	26600	335000	142000	30400	534000	8930	14200	30000
ZINC	(19)	(0.0)	290	(16)	(3.7)	(17)	(16)	(4.0)

PERCENT SOLIDS (%)	NR	NR	NR	NR	NR	NR	NR	NR
OIL AND GREASE (ug/l)	40	16.3	3.15	96.7	10.5	19.1	19.1	172
TOTAL ALKALINITY (ug/l) as CaCO3	5750	1406	907	445	2925	202	21.6	802.4
DILUTED SOLIDS (ug/l)	5	625	23	966	966	966	966	966
SULFATE (ug/l)	390	1751	34.5	96.7	10.5	19.1	19.1	172

FOOTNOTES:

- B: Analyte has been found in the laboratory blank as well as the sample. Indicates possible/probable contamination.
C: Applies to pesticide parameters where the identification has been confirmed by GC/MS.
E: Value is estimated due to presence of interference.
J: An estimated value.
K: Actual value, within the limitations of the method, is less than the given value.
R: Spike sample recovery is not within control limits.
S: Value is determined by standard addition.
D: Duplicate analysis is not within control limits.
E: Sample(s) analyzed at medium concentrations.
C: Correlation coefficient for method of standard addition is less than 0.995.
P: Positive values less than the contract required detection limit.
NR: Not required by contract at this time.

NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
GLACIAL TILL WATER BEARING UNIT
PHASE II SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	NSL85A	NSL95	NSL105	NSL115	NSL14	NSL15	NSL16	NSL18	PAGE 4 OF 4	
Sample Number:	GW0085-02	GW0095-02	GW0105-02	GW0115-02	GW014-02	GW015-02	GW016-02	GW018-02	GW027-02	GW028-02
Sample Type:									FIELD BLANK	FIELD BLANK
Date Sampled:	5-14-85	5-15-85	5-14-85	5-14-85	5-15-85	5-15-85	5-14-85	5-15-85	5-14-85	5-15-85
OIR Number:	ED669	ED617	ED619	ED621	ED625	ED626	ED627	ED628	ED672	ED634
IIR Number:	MED137	MED139	MED141	MED143	MED147	MED148	MED149	MED150	MED505	MED506
.....										
INORGANIC COMPOUNDS (ug/l)										
.....										
ALUMINUM			300							
ANTIMONY										70
ARSENIC					44 R					
BARIUM		400			900					
CALCIUM	51000	210000	207000	70000	204000	81000	68000	184000		
.....										
IRON					22200			2230		
LEAD				30		14	20			
MAGNESIUM	25000	106000	59000	21000	150000	22000	23000	65000		
MANGANESE	50	500	1020	240	600	250	50	4330		
.....										
NICKEL		100			70					
POTASSIUM		11000	6000	[3000]	156000					
SODIUM	25000	360000	75000	28000	495000	9000	13000	41000		
.....										
pH (units)	7.71								8.39	
PERCENT SOLIDS	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
.....										
OIL AND GREASE (ug/l)	2	2	4	3	33	3	4	1		
TOTAL ALKALINITY (mg/l as CaCO3)	525	1640	876	1942	1800	624	572	692	2	4
CHLORIDE (mg/l)	10	694	39	15	809	16	26	71		
DISSOLVED SOLIDS (mg/l)	420	2240	1330	604	3010	524	466	1000	12	34
SULFATE (mg/l)		16	477	77	25	54	77	157		
.....										

FOOTNOTES:

- S: Value determined by method of standard addition.
R: Spike sample recovery is not within control limits.
J: An Estimated Value.
[]: Value is greater than or equal to the instrument detection limit but less than the contract required detection limit.
NR: Not required by contract at this time.

PHASE II SAMPLE TWO REMEDIAL INVESTIGATION REPORT

Sample Location:

Sample Number:	GA001-02	GA002-02	GA003-02	GA004-02	GA005-02	GA006-02	GA007-02	GA008-02	GA009-02-DUP
Sample Type:	5-15-85	5-15-85	5-14-85	5-15-85	5-15-85	5-14-85	5-15-85	5-14-85	5-14-85
Date Sampled:	GA078	GA078	ED668	EB721	EB722	ED613	ED614	ED670	ED671
QIR Number:	ED673	ED673	ED678	ED679	ED679	ED679	ED679	ED679	ED679
QIR Number:	ED673	ED673	ED678	ED679	ED679	ED679	ED679	ED679	ED679

ORGANIC COMPOUNDS (ug/l)

SALES

[illegible]

03-Feb-86

NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
SAND AND GRAVEL WATER BEARING UNIT
PHASE II SAMPLING
REMEDIATION INVESTIGATION REPORT

Sample Location:	MW1	MW2	MW3	MW4	MW5	MW6	MW7	NSL BDA	NSL BDA	PAGE 6 OF 8
Sample Number:	GW001-02	GW002-02	GW003-02	GW004-02	GW005-02	GW006-02	GW007-02	GW0080-02	GW025-02	
Sample Type:									GW0080-02-DUP	
Date Sampled:	5-15-85	5-15-85	5-14-85	5-15-85	5-15-85	5-14-85	5-15-85	5-14-85	5-14-85	
OTR Number:	ED673	EA370	ED668	EB721	EB722	ED613	ED614	ED670	ED671	
ITR Number:	MEC556	MEC557	MEC558	MEC559	MED134	MED135	MED136	MED138	MED503	
.....										
INORGANIC COMPOUNDS (ug/l)										
.....										
ANTIMONY										
ARSENIC								26 R	26 R	
BARIUM	1400									
CALCIUM	111000	53000	42000	23000	36000	64000	73000	36000	38000	
.....										
IRON	4970	350				200				
LEAD				6						
CYANIDE										
MAGNESIUM	122000	17000	21000	7000	13000	29000	27000	18000	19000	
MANGANESE	160		60					40	50	
.....										
NICKEL	120									
POTASSIUM	84000									
SODIUM	440000	37000	38000	72000	44000	22000	18000	36000	34000	
ZINC										
.....										
pH	(units)		8.25					7.76	7.63	
PERCENT SOLIDS	NR	NR	NR	NR	NR	NR	NR	NR	NR	
.....										
OIL AND GREASE (mg/l)										
TOTAL ALKALINITY (mg/l as CaCO3)	1050	330	355	303	264	356	320	307	316	
CHLORIDE (mg/l)	871	7	9	5	4		13	7	9	
DISSOLVED SOLIDS (mg/l)	2400	360	348	306	300	356	394	338	406	
SULFATE (mg/l)	10	13		16			11		11	
.....										

FOOTNOTES:

- S: Value determined by method of standard addition.
- R: Spike sample recovery is not within control limits.
- J: An Estimated Value.
- (): Value is greater than or equal to the instrument detection limit but less than the contract required detection limit.
- NR: Not required by contract at this time.

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NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
SAND AND GRAVEL WATER BEARING UNIT
PHASE II SAMPLING
REMEDIAL INVESTIGATION REPORT

PAGE 7 OF 8

Sample Location:	NSL 90	NSL 90	NSL 100	NSL 110	NSL 12	NSL 13	SBP65	SBP77	
Sample Number: G4089D-02	G4089D-02	G4089D-02	G4089D-02	G4089D-02	G4089D-02	G4089D-02	G4089D-02	G4089D-02	G4089D-02
Sample Type: G4089D-02-DUP	G4089D-02-DUP	G4089D-02-DUP	G4089D-02-DUP	G4089D-02-DUP	G4089D-02-DUP	G4089D-02-DUP	G4089D-02-DUP	G4089D-02-DUP	G4089D-02-DUP
Date Sampled: 5-15-85	5-15-85	5-15-85	5-15-85	5-15-85	5-15-85	5-15-85	5-15-85	5-15-85	5-15-85
OTR Number: ED618	ED618	ED620	ED622	ED624	ED626	ED628	ED630	ED632	ED634
JTR Number: MED140	MED504	MED142	MED144	MED145	MED146	MED501	MED502	MED503	MED506
ORGANIC COMPOUNDS (ug/l)									
VOLATILES									
BENZENE					5 J	1.2 J	120		
CHLOROETHANE					49 J	42 J			
TRICHLOROETHANE					5.6	9.2			
VINYL CHLORIDE					4.7 J	72 J			
ACETONE							110 BJ	10 JB	
2-BUTANONE	11 BJ						92 BJ		
METHYLENE CHLORIDE					2.1 J	4.5 J	6 B	5 JB	7.5
TRANS-1, 2-DICHLOROETHENE					20	130			
1, 1, 1-TRICHLOROETHANE						5.7			
1, 1-DICHLOROETHANE					12	85			
4-METHYL-2-PENTANONE						1.2 J			
ETHYLBENZENE									
TOTAL VOLATILES	0	11	20	22.1	90.4	350.8	328	15	7.5
TOTAL TENTATIVELY IDENTIFIED VOLATILES	0	35.9 J	0	0	0	0	222 J	5.7 J	0
TOTAL PESTICIDES and PCBs	0	0	0	0	0	0	0	0	0
BASE/NEUTRALS and ACIDS									
BENZOIC ACID					7.3 J		2.8 J		
DIETHYLPHTHALATE									
DI-N-OCTYL PHTHALATE									
N-NITROSDI-N-PROPYLAMINE									3.9 J
PHENOL									
DI-N-BUTYL PHTHALATE								10 J	
BIS(2-ETHYLETHYL)PHTHALATE									
TOTAL BASE/NEUTRALS AND ACIDS	0	0	10	10	7.3	0	2.8	10	3.9
TOTAL TENTATIVELY IDENTIFIED BASE/NEUTRALS AND ACIDS	22.4 J	9.5 J	334 J	0 J	801.3 J	284.4 J	2616.3 J	27 J	17.5 J

03-Feb-86

NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
SAND AND GRAVEL WATER BEARING UNIT
PHASE II SAMPLING
REMEDIAL INVESTIGATION REPORT

PAGE 8 OF 8

Sample Locations:	NSL90	NSL90	NSL100	NSL110	NSL12	NSL13	SBP65	SBP77		
Sample Number:	GW0090-02	GW026-02	GW0100-02	GW0110-02	GW012-02	GW013-02	GW065-02	GW077-02	GW027-02	GW028-02
Sample Type:		GW0090-02-DUP							FIELD BLANK	FIELD BLANK
Date Sampled:	5-15-85	5-15-85	5-14-85	5-14-85	5-14-85	5-14-85	5-15-85	5-14-85	5-14-85	5-15-85
QIR Number:	ED618	ED632	ED620	ED622	ED623	ED624	ED629	ED630	ED672	ED634
IR Number:	MED140	MED504	MED142	MED144	MED145	MED146	MED501	MED502	MED505	MED506
.....										
INORGANIC COMPOUNDS (ug/l)										
.....										
ANTIMONY										70
ARSENIC										
BARIUM					870	570	1600			
CALCIUM	95000	97000	27000	41000	107000	150000	207000	56000		
.....										
IRON				16	200	1900	5020			
LEAD										
CYANIDE					60					
MAGNESIUM	40000	40000	11000	19000	140000	89000	160000	25000		
MANGANESE	40	40			370	1030		80		
.....										
NICKEL					110	70				
POTASSIUM	5000	5000			55000	20000	210000			
SODIUM	100000	103000	72000	34000	304000	264000	575000	26000		
ZINC							30 RJ			
.....										
pH									8.39	
PERCENT SOLIDS	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
.....										
OIL AND GREASE (ug/l)										
TOTAL ALKALINITY (mg/l as CaCO3)	744	672	390	422	1500	650	2072	1116	2	4
CHLORIDE (mg/l)	141	196	11	6	916	634	1050	3		
DISSOLVED SOLIDS (mg/l)	862	892	416	432	2660	2010	3060	394	12	34
SULFATE (mg/l)	16	20			15	61	10			
.....										

FOOTNOTES:

S: Value determined by method of standard addition.

R: Spike sample recovery is not within control limits.

J: An Estimated Value.

(): Value is greater than or equal to the instrument detection limit but less than the contract required detection limit.

NR: Not required by contract at this time.

TABLE A-3
MINNESOTA SANITARY LANDFILL LEACHATE RESULTS
REMEDIATION INVESTIGATION REPORT

Page 1 of 2

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TABLE A-3
NORTHSIDE SANITARY LANDFILL LEACHATE RESULTS
REMEDIAL INVESTIGATION REPORT

PAGE 2 OF 2

LEACHATE SAMPLES (LIQUID)				LEACHATE SAMPLES (SOLID)		
Sample Location:		LL001	LL002	LS001	LS002	LS003
Sample Number:		LL001-01	LL002-01	LS001-01	LS002-01	LS003-01
Sample Type:						
Date Sampled:		5-15-85	5-15-85	5-15-85	5-15-85	5-15-85
DTA Number:		ED662	ED663	ED665	ED666	ED667
ITR Number:		MED534	MED535	MED537	MED538	MED539
INORGANIC COMPOUNDS (ug/l)				(ug/kg)		
ALUMINUM	2000	3600		4700	4700	7400
BARIUM		1200				
CALCIUM	67000	660000		64000	70000	49000
CHROMIUM		30		10		
IRON	3400	65000		9100	16000	15000
LEAD	10	42 S		10	9.3	22
MAGNESIUM	12000	160000		19000	22000	14000
MANGANESE		6220		330 JR	460 JR	1400 JR
NICKEL		120				
POTASSIUM		310000				
SODIUM	6000	345000				
VANADIUM						
ZINC		50 JR		30	30	63
pH (units)						
PERCENT SOLIDS (K) (INORGANICS)	NR	NR	NR	76	85	87
OIL AND GREASE (mg/l)		134				22000
TOTAL ALKALINITY(mg/l as CaCO3)	115	1990	4	NR	NR	NR
CHLORIDE(mg/l)	13	87		NR	NR	NR
DISSOLVED SOLIDS(mg/l)	316	5530	20	NR	NR	NR
SULFATE(mg/l)	104	73	13	NR	NR	NR

FOOTNOTES:

- B: Analyte has been found in the laboratory blank as well as the sample. Indicates possible/probable contamination.
 J: An Estimated Value.
 R: Spike sample recovery is not within control limits.
 S: Value is determined by standard addition.
 I: Positive values less than the contract required detection limit.
 **: Sample(s) analyzed at medium concentrations.
 NR: Not required by contract at this time.

Note: All concentrations and quantitation limits are reported on a dry weight basis (surface soil, sediment and boring samples only).
 Note: ED663 Acid data unacceptable.

TABLE 9
NORTHSIDE SANITARY LANDFILL
LIQUID LEACHATE RESULTS
PHASE III - SAMPLING
REMEDIATION INVESTIGATION
LIQUID LEACHATE

Sample Location:	LEACHATE TANK 1	LEACHATE TANK 1	LEACHATE TANK 2	LEACHATE TANK 3	PAGE 1 OF 2
Sample Number:	NSL-LL005-02	NSL-LL007-02	NSL-LL004-02	NSL-LL004-02	NSL-LL008-02
Sample Type:		DUP NSL-LL005-02			FIELD BLANK
Date Sampled:	11/21/85	11/21/85	11/21/85	11/21/85	11/21/85
OTA Number:	EE365	EE367	EE366	EE364	EE368
IR Number:	ME6200	ME602	ME601	ME619	ME603
ORGANIC COMPOUNDS (ug/l)					
VOLATILES					
BENZENE	5	9 J		7	
CHLOROBENZENE				6	
1, 1-DICHLORoETHANE			460		
TRANS-1, 2-DICHLORoETHENE			1300		
ETHYL BENZENE	100	220		84	
METHYLENE CHLORIDE			2200	7	
TOLUENE	21	23		35	
ACETONE			11000 J	120 J	J
2-BUTANONE	15		12000	95	
4-METHYL-2-PENTANONE	11		2000	72	
TOTAL HYDROCARBONS	540	1100	11000	400	
TOTAL VOLATILES	692	1352	39960	926	
TOTAL TENTATIVELY IDENTIFIED VOLATILES	542	700	706	330	8
BASE NEUTRALS and ACIDS					
4-CHLORO-3-METHYLPHENOL				15 J	
2, 4-DIMETHYLPHENOL		13 J			
PHENOL	7 J	15 J	370 J		
BENZOIC ACID			1460 J	180 J	
4-METHYLPHENOL			1350 J	37 J	
ISOPHTHALIC ACID				73 J	
NAPHTHALENE	23 J	16 J			
BIS(2-ETHYLHEXYL)PHTHALATE	24 J	64 J	650 J	20 J	
DI-N-BUTYL PHTHALATE				3 J	
DIETHYL PHTHALATE	24 J	21 J	71 J	27 J	
TOTAL BASE NEUTRALS and ACIDS	70 J	129 J	3901 J	355 J	
TOTAL TENTATIVELY IDENTIFIED ACIDS BASE/NEUTRALS	4149	2439	10270	1844	15
PESTICIDES and PCBs					
UNUSABLE					
TOTAL PESTICIDES and PCBs	0	0	0	0	

TABLE 9
NORTHSIDE SANITARY LANDFILL
LIQUID LEACHATE RESULTS
PHASE III - SAMPLING
REMEDIAL INVESTIGATION
LIQUID LEACHATE

Sample Locations:	LEACHATE TANK 1	LEACHATE TANK 1	LEACHATE TANK 2	LEACHATE TANK 3	PAGE 2 OF 2
Sample Numbers:	MSL-LL003-02	MSL-LL007-02	MSL-LL006-02	MSL-LL004-02	MSL-LL008-02
Sample Types:		DUP MSL-LL005-02			FIELD BLANK
Date Sampled:	11/21/85	11/21/85	11/21/85	11/21/85	11/21/85
QTR Numbers:	EE365	EE367	EE366	EE364	EE368
IR Numbers:	ME6200	ME602	ME601	ME199	ME603
=====					
INORGANIC COMPOUNDS (ug/l)					
=====					
ALUMINUM	(133)	(183)	334	(74)	
ARSENIC			11		
BARIUM	782	732	(117)	349	
CALCIUM	152000	159000	262000	219000	
CHROMIUM	15	16	14	10	
COBALT	(11)	(9.7)	(11)	(12)	
COPPER	(14)	(14)	(21)	28	(7.8)
IRON	21400	24300	36800	44100	(41)
LEAD	39	31	28	22	8.7
MAGNESIUM	175000	176000	135000	88900	
MANGANESE	185	223	827	731	
NICKEL	99	101	58	(39)	
POTASSIUM	332000	331000	212000	145000	
SILVER			(5.1)	(3.8)	
SODIUM	629000	623000	385000	206000	
VANADIUM	(4.7)	(5.2)	(7.1)		
ZINC	271	108	149	157	(7.4)
=====					
(ug/l)					
OIL AND GREASE	<5	<5	37	<5	<5
=====					

FOUNDTEN 1: Estimated value.
 0: Used when the analyte is found in the laboratory sample.
 Indicates possible/probable contamination.
 2: Coelution with other compounds prevents spectral
 confirmation according to contact guidelines; this
 compound is believed to be present.
 3: Positive value less than the contract required
 detection limit.

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TABLE B-1. MSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - METALS

UNFILTERED						FILTERED						
Sample Number:	LT01-01	DLT01-01	LT01-02	LT01-03	LT01-04	LT01-05	LT01-01	DLT01-01	LT01-02	LT01-03	LT01-04	LT01-05
Sample Location:	MSLLT J	MSLLT J	MSLLT J	MSLLT J	MSLLT J	MSLLT J	MSLLT J	MSLLT J	MSLLT J	MSLLT J	MSLLT J	MSLLT J
Date Sampled:	8-24-87	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87	8-24-87	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87
ITR Number:	MEB292	MEB297	MEB303	MEC782	MEB312	MEK345	ME1705	ME1706	ME1716	ME1723	ME6986	ME6996
INORGANICS UG/L												
Aluminum	---	---	---	(67)	(56)	(62)	(56)	(56)	(66)	(49)	(45)	(39)
Antimony	---	---	---	---	---	---	71	---	---	---	---	---
Arsenic	11	12	(10)	---	---	11	---	---	---	---	---	---
Barium	541	549	553	545	543	518	398	404	404	394	386	410
Beryllium	---	---	---	---	---	---	---	---	---	---	---	---
Cadmium	---	---	---	---	---	---	(4.2)	---	---	---	---	---
Calcium	124000	126000	127000	126000	126000	118000	124000	124000	124000	118000	116000	120000
Chromium	16 BJ	14 BJ	13 BJ	16 BJ	18 BJ	29 BJ	22 BJ	21 BJ	20 BJ	19 BJ	17 BJ	20 BJ
Cobalt	---	---	---	---	---	---	(11)	---	---	(11)	(11)	---
Copper	(6.1) B	(6.2) B	---	---	---	---	(7.4) B	(6.6) B	---	---	---	---
Iron	22000	22200	22700	22500 J	22600 J	21500	2920 J	3130 J	2880 J	2420	2450	4070
Lead	16 BSJ	17 BSJ	20 BSJ	27 BSJ	26 BSJ	17 BSJ	9.1 BSJ	10 BSJ	12 BSJ	8.4 BSJ	9 BSJ	---
Magnesium	177000	179000	181000	175000	176000	168000	178000	179000	180000	173000	172000	174000
Manganese	87	86	88	89	91	87	85	85	89	76	75	79
Mercury	---	---	---	---	---	---	---	---	---	---	---	---
Nickel	74	78	82	74	70	76	77	81	81	78	78	74
Potassium	403000	406000	409000	404000	402000	369000	391000	392000	393000	397000	395000	403000
Selenium	---	---	---	---	---	---	---	8 SJ	---	---	---	---
Silver	---	---	---	---	---	---	---	---	---	---	---	---
Sodium	691000	697000	706000	686000	687000	638000	695000	697000	702000	684000	679000	693000
Thallium	---	---	---	---	---	---	---	---	---	---	---	---
Tin	---	---	---	---	---	---	192 BJ	---	---	---	---	---
Vanadium	---	---	---	---	---	---	(12)	---	---	---	---	---
Zinc	62	62	101	108	143	162	42	44	64	66	96	102
Cyanide	---	---	---	---	---	---	NA	NA	NA	NA	NA	NA

UNFILTERED						FILTERED						
Sample Number:	LT02-01	LT02-02	LT02-03	DLT02-03	LT02-04	LT02-05	LT02-01	LT02-02	LT02-03	DLT02-03	LT02-04	LT02-05
Sample Location:	MSLLT 2	MSLLT 2	MSLLT 2	MSLLT 2	MSLLT 2	MSLLT 2	MSLLT 2	MSLLT 2	MSLLT 2	MSLLT 2	MSLLT 2	MSLLT 2
Date Sampled:	8-24-87	8-25-87	8-26-87	8-26-87	8-27-87	8-28-87	8-24-87	8-25-87	8-26-87	8-26-87	8-27-87	8-28-87
ITR Number:	MEB293	MEB304	MEC783	MEC787	MEB313	MEK472	ME1707	ME1717	ME1722	ME1725	ME6987	ME6997
INORGANICS UG/L												
Aluminum	(90)	(73)	(150)	(135)	(143)	(140)	(58)	(52)	(47)	(46)	(66)	(40)
Antimony	---	---	---	---	---	---	---	---	---	---	---	---
Arsenic	12 S	12	11	12	12	13	---	---	---	11	---	---
Barium	235	239	242	234	235	233	(158)	(172)	(175)	(175)	(174)	(173)
Beryllium	---	---	---	---	---	---	---	---	---	---	---	---
Cadmium	---	---	---	---	---	---	---	---	---	---	---	---
Calcium	200000	205000	205000	200000	200000	197000	200000	196000	190000	188000	188000	194000
Chromium	15 BJ	17 BJ	21 BJ	21 BJ	18 BJ	19 BJ	17 BJ	20 BJ	25 BJ	24 BJ	22 BJ	24 BJ
Cobalt	---	---	---	---	---	---	---	---	---	---	---	---
Copper	(7) B	---	---	---	---	---	(8.3) B	---	---	---	---	---
Iron	29700	30300	30100 J	29500 J	29400 J	29100	3280 J	9590 J	10100	9980	11700	9620
Lead	15 BSJ	18 BSJ	25 BSJ	22 BSJ	22 BSJ	14 BSJ	16 BSJ	6.5 BSJ	9.9 BSJ	9.5 BSJ	8.5 BSJ	---
Magnesium	181000	185000	182000	177000	178000	179000	189000	182000	178000	175000	176000	179000
Manganese	241	247	248	241	243	243	234	236	220	221	218	221
Mercury	---	---	---	---	---	---	---	---	---	---	---	---
Nickel	92	95	92	89	90	91	98	86	91	92	92	90
Potassium	306000	312000	311000	301000	302000	294000	309000	294000	304000	298000	301000	309000
Selenium	---	---	---	---	---	---	---	---	---	---	---	---
Silver	---	---	---	---	---	---	---	---	---	---	---	---
Sodium	603000	619000	611000	592000	595000	587000	634000	608000	603000	594000	602000	617000
Thallium	---	---	---	---	---	---	---	---	---	---	---	---
Tin	---	---	---	---	---	---	---	(26) B	---	---	---	---
Vanadium	---	---	(7.4)	---	---	---	---	---	---	---	---	---
Zinc	73	81	119	118	129	155	25 B	(20) B	24 B	28 B	31 B	24 B
Cyanide	---	---	---	---	---	---	NA	NA	NA	NA	NA	NA

NOTE: B: Analyte has been found in the laboratory or field blank as well as the sample.
Indicates probable/possible contamination.

[]: Positive values less than the contract required detection limit.

S: Value is determined by standard addition.

J: An estimated value.

NA: Not analyzed.

NS: Not sampled.

---: Not detected.

TABLE D-1. MSL/FECF PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - METALS

Sample Number:	HMMSL12-01	HMMSL12-02	HMMSL12-03	HMMSL12-04	HMMSL12-05	HMMSL12-01	HMMSL12-02	HMMSL12-03	HMMSL12-04	HMMSL12-05	Sample Number:	HMMSL12-01	HMMSL12-02	HMMSL12-03	HMMSL12-04	HMMSL12-05
Date Sampled:	8-24-87	8-24-87	8-24-87	8-24-87	8-24-87	8-24-87	8-24-87	8-24-87	8-24-87	8-24-87	Date Sampled:	8-24-87	8-24-87	8-24-87	8-24-87	8-24-87
Lab Number:	MEB290	MEB301	MEC791	MEK337	MEK343	ME1704	ME1712	ME1730	ME6984	ME6994	Lab Number:	MEB290	MEB301	MEC791	MEK337	MEK343
Sample Location:	MSL12	MSL12	MSL12	MSL12	MSL12	MSL12	MSL12	MSL12	MSL12	MSL12	Sample Location:	MSL12	MSL12	MSL12	MSL12	MSL12
Aluminum	56100	14700	31200	13200	18100	77500	19100	1020	260	61000	25100	19100	260	61000	69800	70400
Arsenic	29	19	18	20	18	32	1700	5.3	5.3	968	968	968	968	968	968	968
Barium	1500	1200	1350	1270	1210	1020	1020	1020	1020	968	968	968	968	968	968	968
Beryllium	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Cadmium	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Calcium	322000	207000	358000	354000	202000	723000	74100	74100	61000	69800	70400	71900	71900	70400	70400	70400
Chromium	95	42	94	94	51	185	80	486	486	11	390	11	390	11	390	11
Cobalt	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Copper	204	56800	30500	30500	74100	286000	67	67	251	390	390	390	390	390	390	390
Iron	132000	119000	119000	119000	119000	119000	119000	119000	119000	119000	119000	119000	119000	119000	119000	119000
Lead	66	38	38	38	38	38	38	38	38	38	38	38	38	38	38	38
Manganese	211000	174000	1560	1560	175000	35400	3440	0.3	358	125000	125000	125000	125000	125000	125000	125000
Mercury	1470	789	1470	1470	873	144	144	144	144	131	131	131	131	131	131	131
Nickel	213	130	192	192	148	82	82	82	82	76	76	76	76	76	76	76
Potassium	98100	93800	90400	90400	97600	88800	88800	88800	88800	89000	89000	89000	89000	89000	89000	89000
Silver	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Sodium	382000	388000	393000	388000	393000	392000	31300	31300	31300	384000	384000	394000	394000	384000	394000	394000
Thallium	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Vanadium	107	100	100	100	60	233	233	233	233	233	233	233	233	233	233	233
Zinc	764	620	1400	1400	441	2160	25	25	25	35	35	35	35	35	35	35
Cyanide	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---

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TABLE B-1. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - METALS

UNFILTERED								FILTERED							
Sample Number: MWNSL13-01	MWNSL13-02	DMWNSL13-02	MWNSL13-03	DMWNSL13-03	MWNSL13-04	MWNSL13-05	MWNSL13-01	MWNSL13-02	DMWNSL13-02	MWNSL13-03	DMWNSL13-03	MWNSL13-04	MWNSL13-05		
Sample Location: NSLAW 13	NSLAW 13	DMNSLAW 13	NSLAW 13	DMNSLAW 13	NSLAW 13	NSLAW 13	NSLAW 13	NSLAW 13	DMNSLAW 13	NSLAW 13	DMNSLAW 13	NSLAW 13	NSLAW 13		
Date Sampled: 8-24-87	8-25-87	8-25-87	8-26-87	8-26-87	8-27-87	8-28-87	8-24-87	8-25-87	8-25-87	8-26-87	8-26-87	8-27-87	8-28-87		
ITR Number: NEB299	NEB299	NEB306	NEC779	NEC785	NEC789	NEC342	NE1713	NE1713	NE1714	NE1729	NE1728	MEG982	MEG993		
INORGANICS UG/L															
Aluminum	NS	[41]	---	[166]	[180]	447	10900	NS	[16]	[18]	[17]	[23]	[18]	---	---
Antimony	NS	---	---	---	---	---	---	NS	---	---	---	---	---	---	---
Arsenic	NS	18	17	15 S	14 S	12	14	NS	16	14	---	---	18 S	12	---
Barium	NS	457	446	451	461	451	485	NS	440	435	366	371	998	425	---
Beryllium	NS	---	---	---	---	---	---	NS	---	---	---	---	---	---	---
Cadmium	NS	---	---	---	---	---	6.4 J	NS	---	---	---	---	---	---	---
Calcium	NS	96300	95300	91800	93000	92600	189000	NS	96900	95800	77900	79200	99200	86800	---
Chromium	NS	---	(4.5) B	(6.7) B	(5) B	(4.6) B	49 BJ	NS	(8.6) B	(9.4) B	(7.4) B	(8.1) B	10 BJ	(4.5) B	---
Cobalt	NS	---	---	---	---	---	(15)	NS	---	---	---	---	---	---	---
Copper	NS	---	---	(6.8) B	---	---	44	NS	(6.3) B	---	---	---	---	---	---
Iron	NS	6580	6190	6110 J	6520 J	6500 J	33900	NS	3800 J	4000 J	1370 J	523 J	1320	2710	---
Lead	NS	---	---	---	---	10 BSJ	45 SJ	NS	---	---	---	5.8 BSJ	9.2 BSJ	---	---
Magnesium	NS	52400	51700	50900	50300	51300	89300	NS	52900	52400	44500	45100	114000	47400	---
Manganese	NS	525	516	454	462	504	1070	NS	529	522	382	386	86	400	---
Mercury	NS	---	---	---	---	---	---	NS	---	---	---	---	---	---	---
Nickel	NS	(25)	(24)	(20)	(21)	(25)	76	NS	(24)	(27)	(20)	(24)	68	(17)	---
Potassium	NS	30100	29700	31600	30500	28800	28800	NS	29500	29100	27000	27400	93900	27500	---
Selenium	NS	---	---	---	---	---	---	NS	---	---	---	---	---	---	---
Silver	NS	---	---	---	---	---	---	NS	---	---	---	---	---	---	---
Sodium	NS	104000	102000	97700	97000	94400	92200	NS	109000	108000	89800	91600	413000	88800	---
Thallium	NS	---	---	---	---	---	---	NS	---	---	---	---	---	---	---
Tin	NS	---	---	---	---	---	---	NS	---	---	---	---	---	---	---
Vanadium	NS	---	---	---	---	---	---	NS	---	---	---	---	---	---	---
Zinc	NS	(3.2) B	---	(9.1) B	(8.2) B	(14) B	255	NS	(9.2) B	(4.7) B	(7.3) B	(13) B	55	(9.9) B	---
Cyanide	NS	---	---	---	---	---	---	NS	NA	NA	NA	NA	NA	NA	---

UNFILTERED								FILTERED							
Sample Number: MWNSLBSA-01	DMWNSLBSA-01	MWNSLBSA-02	MWNSLBSA-03	MWNSLBSA-04	MWNSLBSA-05	MWNSLBSA-01	DMWNSLBSA-01	MWNSLBSA-02	MWNSLBSA-03	MWNSLBSA-04	MWNSLBSA-05				
Sample Location: NSLAW BSA	DMNSLAW BSA	NSLAW BSA	NSLAW BSA	NSLAW BSA	NSLAW BSA	NSLAW BSA	DMNSLAW BSA	NSLAW BSA	NSLAW BSA	NSLAW BSA	NSLAW BSA				
Date Sampled: 8-24-87	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87	8-24-87	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87				
ITR Number: NEB286	NEB295	NEB298	NEB309	NEC788	NEK340	NE1701	NE1703	NE1710	NE1726	MEG981	MEG991				
INORGANICS UG/L															
Aluminum	528	469	(26)	1490	546	717	(74)	(67)	(71)	---	---	---	---	---	---
Antimony	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Arsenic	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Barium	256	262	279	289	286	286	238	241	983	229	238	237	---	---	---
Beryllium	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Cadmium	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Calcium	58700	60600	60800	67000	63300	65400	54000	55600	72300	56800	56500	56500	---	---	---
Chromium	---	(4.2) B	---	(5.9) B	(5.8) B	---	(5.6) B	(5.6) B	(7.8) B	(8.9) B	---	---	---	---	---
Cobalt	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Copper	---	(7.2) B	---	(7.2) B	---	---	(8.5) B	(11) B	---	---	---	---	---	---	---
Iron	2330	2370	1330	3710	1690 J	2640	1500 J	1570 J	1320 J	(53) B	(53) B	139	---	---	---
Lead	---	---	4.5 BJ	---	---	---	---	13 BJ	5.7 BSJ	---	---	5.6 BJ	---	---	---
Magnesium	28400	28700	28700	30300	30000	30600	26400	27300	127000	28200	27600	27400	---	---	---
Manganese	55	52	34	85	56	84	41	53	144	28 B	29 B	45	---	---	---
Mercury	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Nickel	---	---	---	---	---	---	---	---	79	---	---	---	---	---	---
Potassium	(1630)	(1570)	(1450)	(2030)	(1650)	(1920)	(1400)	(1480)	86100	(1910)	(1640)	(1760)	---	---	---
Selenium	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Silver	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Sodium	27100	27000	25700	27300	27200	26600	25000	27700	384000	34100	32300	31200	---	---	---
Thallium	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Tin	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Vanadium	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Zinc	(9) B	(17) B	(3.2) B	(19) B	23 B	28 B	(10) B	(16) B	32 B	(6.3) B	(4.3) B	(5.5) B	---	---	---
Cyanide	---	---	---	---	---	---	NA	NA	NA	NA	NA	NA	---	---	---

TABLE B-1. MSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - METALS

UNFILTERED										FILTERED									
Sample Number:	HECMA-01	HECMA-02	HECMA-03	HECMA-04	HECMA-05	HECMA-01	HECMA-02	HECMA-03	HECMA-04	HECMA-05	HECMA-01	HECMA-02	HECMA-03	HECMA-04	HECMA-05	HECMA-01	HECMA-02	HECMA-03	HECMA-04
Date Sampled:	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87	8-24-87	8-25-87	8-26-87	8-27-87
ITR Number:	MEB300	MEB310	MEC790	MEK341	MEK341	ME1711	ME1727	ME6983	ME6992	ME6995	ME1711	ME1727	ME6983	ME6992	ME6995	ME1711	ME1727	ME6983	ME6992
Sample Location:	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A	ECCMA 3A
Aluminum	19000	3960	1150	23200	23200	NS	[75]	[52]	---	---	NS	---	---	---	---	NS	---	---	---
Arsenic	20	21	26	29	29	NS	19	16	---	---	NS	---	---	---	---	NS	---	---	---
Barium	1240	1180	1170	1180	1180	NS	1390	965	---	---	NS	---	---	---	---	NS	---	---	---
Beryllium	---	---	[2]	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Cadmium	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Calcium	316000	233000	274000	298000	298000	NS	124000	98600	---	---	NS	---	---	---	---	NS	---	---	---
Chromium	30	20	[6.2]	49	49	NS	11	12	---	---	NS	---	---	---	---	NS	---	---	---
Cobalt	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Copper	152	137	44	172	172	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Iron	82100	38800	93900	93900	93900	NS	4460	2480	---	---	NS	---	---	---	---	NS	---	---	---
Lead	57	24	54	54	54	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Magnesium	194000	166000	181000	181000	181000	NS	124000	114000	---	---	NS	---	---	---	---	NS	---	---	---
Manganese	1420	825	1160	1500	1500	NS	91	84	---	---	NS	---	---	---	---	NS	---	---	---
Mercury	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Nickel	140	95	74	148	148	NS	70	68	---	---	NS	---	---	---	---	NS	---	---	---
Potassium	99000	98000	95200	92400	92400	NS	90800	93700	---	---	NS	---	---	---	---	NS	---	---	---
Selenium	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Silver	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Sodium	415000	418000	419000	483000	483000	NS	426000	98900	---	---	NS	---	---	---	---	NS	---	---	---
Thallium	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Tin	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Vanadium	35	[23]	71	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Zinc	280	---	602	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Cyanide	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Aluminum	92000	36300	20800	13000	13000	NS	376	---	---	---	NS	---	---	---	---	NS	---	---	---
Arsenic	45	32	20	14	14	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Beryllium	4120	3960	3920	4080	4080	NS	3640	---	---	---	NS	---	---	---	---	NS	---	---	---
Cadmium	5.1	[1.9]	[1.5]	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Calcium	917000	568000	376000	329000	329000	NS	260000	250000	---	---	NS	---	---	---	---	NS	---	---	---
Chromium	154	87	56	44	44	NS	14	12	---	---	NS	---	---	---	---	NS	---	---	---
Cobalt	239	108	60	36	36	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Copper	195000	90800	55600	40300	40300	NS	13000	6950	---	---	NS	---	---	---	---	NS	---	---	---
Iron	186	62	27	18	18	NS	6.1	5.4	---	---	NS	---	---	---	---	NS	---	---	---
Lead	338000	234000	185000	170000	170000	NS	150000	146000	---	---	NS	---	---	---	---	NS	---	---	---
Magnesium	1960	1700	867	534	534	NS	77	58	---	---	NS	---	---	---	---	NS	---	---	---
Manganese	0.3	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Mercury	265	135	93	76	76	NS	43	181	---	---	NS	---	---	---	---	NS	---	---	---
Nickel	79200	78800	75900	73500	73500	NS	66600	68100	---	---	NS	---	---	---	---	NS	---	---	---
Potassium	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Selenium	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Silver	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Sodium	398000	432000	421000	419000	419000	NS	421000	421000	---	---	NS	---	---	---	---	NS	---	---	---
Thallium	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Tin	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Vanadium	187	82	[48]	[33]	[33]	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Zinc	780	456	224	133	133	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Cyanide	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Aluminum	92000	36300	20800	13000	13000	NS	376	---	---	---	NS	---	---	---	---	NS	---	---	---
Arsenic	45	32	20	14	14	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Beryllium	4120	3960	3920	4080	4080	NS	3640	---	---	---	NS	---	---	---	---	NS	---	---	---
Cadmium	5.1	[1.9]	[1.5]	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Calcium	917000	568000	376000	329000	329000	NS	260000	250000	---	---	NS	---	---	---	---	NS	---	---	---
Chromium	154	87	56	44	44	NS	14	12	---	---	NS	---	---	---	---	NS	---	---	---
Cobalt	239	108	60	36	36	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Copper	195000	90800	55600	40300	40300	NS	13000	6950	---	---	NS	---	---	---	---	NS	---	---	---
Iron	186	62	27	18	18	NS	6.1	5.4	---	---	NS	---	---	---	---	NS	---	---	---
Lead	338000	234000	185000	170000	170000	NS	150000	146000	---	---	NS	---	---	---	---	NS	---	---	---
Magnesium	1960	1700	867	534	534	NS	77	58	---	---	NS	---	---	---	---	NS	---	---	---
Manganese	0.3	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Mercury	265	135	93	76	76	NS	43	181	---	---	NS	---	---	---	---	NS	---	---	---
Nickel	79200	78800	75900	73500	73500	NS	66600	68100	---	---	NS	---	---	---	---	NS	---	---	---
Potassium	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Selenium	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Silver	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Sodium	398000	432000	421000	419000	419000	NS	421000	421000	---	---	NS	---	---	---	---	NS	---	---	---
Thallium	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Tin	---	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Vanadium	187	82	[48]	[33]	[33]	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Zinc	780	456	224	133	133	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---
Cyanide	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	NS	---	---	---

UNFILTERED										FILTERED									
Sample Number:	MMF8-01	MMF8-02	MMF8-03	MMF8-04	MMF8-05	MMF8-01	MMF8-02	MMF8-03	MMF8-04	MMF8-05	Sample Number:	MMF8-01	MMF8-02	MMF8-03	MMF8-04	MMF8-05			
MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB	MSLNM FB			
Date Sampled:	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87	8-28-87			
IR Number:	NEB296	NEB307	NE1732	NEK338		NE1704	NE1720	NE6868	NE5900		NE1704	NE1720	NE6868	NE5900					
INORGANICS UG/L																			
Aluminum	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Antimony	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Arsenic	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Barium	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Beryllium	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Cadmium	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Calcium	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Chromium	(4.9)	---	14	---	NS	(4.9)	(7.6)	18	---	NS	---	---	---	---	---	NS			
Cobalt	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Copper	---	---	(6) B	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Iron	---	---	(45)	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Lead	---	---	---	(34)	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Magnesium	---	---	---	5.6	NS	5.4 J	---	---	---	NS	---	---	---	---	---	NS			
Manganese	---	---	---	---	NS	(4.7)	(4.8)	---	---	NS	---	---	---	---	---	NS			
Mercury	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Nickel	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Potassium	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Selenium	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Silver	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Sodium	---	---	(2580)	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Sulfur	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Thallium	---	---	---	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Tin	---	---	87	---	NS	---	---	---	---	NS	---	---	---	---	---	NS			
Vanadium	---	(6)	(7.9) B	(4.1) B	NS	(7.2) B	---	(3.6) B	---	NS	---	---	---	---	(4.7) B	NS			
Zinc	---	---	---	---	NS	NA	NA	NA	NA	NS	---	---	---	---	NA	NS			

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TABLE B-2. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - ORGANIC RESULTS

Sample Number:	LT01-01	DLT01-01	LT01-02	LT01-03	LT01-04	LT01-05
Sample Location:	NSLLT 1	NSLLT 1	NSLLT 1	NSLLT 1	NSLLT 1	NSLLT 1
Date Sampled:	8/24/87	8/24/87	8/25/87	8/26/87	8/27/87	8/28/87
OTR Number:	EL304	EL509	EL515	EL540	EL517	EL528
=====						
VOLATILE ORGANICS: ug/l						
Chloromethane	---	---	---	---	---	---
Bromomethane	---	---	---	---	---	---
Vinyl Chloride	---	---	---	---	---	---
Chloroethane	---	---	---	---	---	---
Methylene Chloride	---	---	---	---	100 J	100 J
Acetone	---	65 J	71 J	52 J	370 J	430 J
Carbon Disulfide	---	6 J	6 J	---	---	---
1,1-Dichloroethene	---	---	---	---	---	---
1,1-Dichloroethane	---	---	---	---	---	---
1,2-Dichloroethene (Total)	---	---	---	---	---	---
Chloroform	---	---	---	---	---	---
1,2-Dichloroethane	---	---	---	---	---	---
2-Butanone	300 J	200 J	240 J	---	180 J	---
1,1,1-Trichloroethane	---	---	---	---	---	---
Carbon Tetrachloride	---	---	---	---	---	---
Vinyl Acetate	---	---	---	---	---	---
Bromodichloromethane	---	---	---	---	---	---
1,2-Dichloropropane	---	---	---	---	---	---
cis-1,3-Dichloropropene	---	---	---	---	---	---
Trichloroethene	---	---	---	---	---	---
Dibromochloromethane	---	---	---	---	---	---
1,1,2-Trichloroethane	---	---	---	---	---	---
Benzene	---	---	---	16 J	---	---
cis-1,3-Dichloropropene	---	---	---	---	---	---
Trans-1,2-Dichloropropene	---	---	---	---	---	---
Bromoform	---	---	---	---	---	---
4-Methyl-2-Pentanone	---	---	---	---	---	---
2-Hexanone	---	---	---	---	---	---
Tetrachloroethene	---	---	---	---	---	---
1,1,2,2-Tetrachloroethane	---	---	---	---	---	---
Toluene	38 J	25 J	25 J	21 J	58 J	63 J
Chlorobenzene	---	---	---	---	---	---
Ethylbenzene	---	---	---	---	69 J	550 J
Styrene	---	---	---	---	---	---
Total Iylenes	450 J	190 J	1200 J	1900 J	6000 J	6500 J
=====						
SEMIVOLATILE ORGANICS						
Phenol	11 J	---	---	5 J	---	---
Benzyl Alcohol	---	---	---	2 J	---	---
1,2-Dichlorobenzene	---	---	---	---	---	---
4-Methylphenol	---	---	---	4 J	---	---
2,4-Dimethylphenol	---	---	---	36 J	---	---
Benzoic Acid	60 J	---	---	---	---	---
Naphthalene	---	15 J	17	---	5 J	12 J
4-Chloro-3-Methylphenol	---	13 J	---	14	---	15 J
2-Methylnaphthalene	---	---	---	---	---	---
Diethylphthalate	---	13 J	---	---	---	---
Di-n-Butylphthalate	---	3 JB	3 JB	2 JB	---	---
bis(2-Ethylhexyl) Phthalate	---	4 J	4 J	---	---	---
3-Nitroaniline	---	---	---	---	---	---
4-Nitroaniline	---	---	---	---	---	---

NOTE: B: Analyte has been found in the field blank as well
as the sample. Indicates probable/possible contamination.
NS: Not sampled.
NR: Not reported.
---: Not detected.

S: Value is determined by standard addition.
J: An estimated value or the report value
is less than the contract required
quantification limit.
R: Unusable, indicates possible false negative.

TABLE B-2. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - ORGANIC RESULTS

Sample Number:	Sample Location:	Date Sampled:	OTR Number:	NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
L102-01	NSLL1 2	8/24/87	EL505	
L102-02	NSLL1 2	8/25/87	EL530	
L102-03	NSLL1 2	8/26/87	EL541	
DL102-03	NSLDL1 2	8/26/87	EL545	
L102-04	NSLL1 2	8/27/87	EL518	
L102-05	NSLL1 2	8/28/87	EL529	
VOLATILE ORGANICS: ug/l				
Chloroethane	---	---	---	---
Bromoethane	---	---	---	---
Vinyl Chloride	---	---	---	---
Chloroethane	130 J	---	---	---
Methylene Chloride	---	---	---	---
Acetone	83 J	---	---	---
Carbon Disulfide	5 J	---	---	---
1,1-Dichloroethane	---	---	---	---
1,1-Dichloroethane	---	---	---	---
1,2-Dichloroethane (Total)	7 J	---	---	---
Chloroform	---	---	---	---
1,2-Dichloroethane	---	---	---	---
2-Butanone	220 J	---	---	---
1,1,1-Trichloroethane	---	---	---	---
Carbon Tetrachloride	---	---	---	---
Vinyl Acetate	---	---	---	---
Bromodichloroethane	---	---	---	---
1,2-Dichloropropane	---	---	---	---
CIS-1,3-Dichloropropane	---	---	---	---
Trichloroethene	---	---	---	---
Dibromochloroethane	---	---	---	---
1,1,2-Trichloroethane	---	---	---	---
Benzene	120 J	---	---	---
CIS-1,3-Dichloropropane	---	---	---	---
TRANS-1,2-Dichloropropane	---	---	---	---
Bromoforn	---	---	---	---
4-Methyl-2-Pentanone	11 J	---	---	---
2-Hexanone	37 J	---	---	---
1,1,2,2-Tetrachloroethane	---	---	---	---
Toluene	24 J	---	---	---
Chlorobenzene	---	---	---	---
Ethylbenzene	---	---	---	---
Styrene	---	---	---	---
Total Xylenes	390 J	---	---	---
SEMIVOLATILE ORGANICS				
Phenol	---	---	---	---
Benzyl Alcohol	---	---	---	---
1,2-Dichlorobenzene	14 J	---	---	---
4-Methylphenol	---	---	---	---
2,4-Dimethylphenol	91	---	---	---
Benzoic Acid	---	---	---	---
Naphthalene	8 J	---	---	---
4-Chloro-3-Methylphenol	---	---	---	---
2-Methylnaphthalene	---	---	---	---
Dichlorophthalate	25 J	---	---	---
Di-n-Butylphthalate	2 BJ	---	---	---
Bis(2-Ethylhexyl) Phthalate	8 J	---	---	---
3-Mitroaniline	---	---	---	---
4-Mitroaniline	---	---	---	---

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TABLE B-2. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - ORGANIC RESULTS

Sample Number:	LT03-01	LT03-02	LT03-03	LT03-04	LT03-05
Sample Location:	NSLLT 3	NSLLT 3	NSLLT 3	NSLLT 3	NSLLT 3
Date Sampled:	8/24/87	8/25/87	8/26/87	8/27/87	8/28/87
DTR Number:	EL506	EL531	EL542	EL519	EL550
VOLATILE ORGANICS: ug/l					
Chloroethane	---	---	---	---	---
Bromoethane	---	---	---	---	---
Vinyl Chloride	---	---	---	---	13 J
Chloroethane	---	---	---	---	---
Methylene Chloride	---	---	---	---	---
Acetone	---	---	---	4 J	---
Carbon Disulfide	---	---	---	---	---
1,1-Dichloroethene	---	---	---	---	---
1,1-Dichloroethane	---	---	---	---	47 J
1,2-Dichloroethene (Total)	---	---	---	---	38 J
Chloroform	---	---	---	---	---
1,2-Dichloroethane	---	---	---	---	---
2-Butanone	---	---	---	---	---
1,1,1-Trichloroethane	---	---	---	---	---
Carbon Tetrachloride	---	---	---	---	---
Vinyl Acetate	---	---	---	---	---
Bromodichloroethane	---	---	---	---	---
1,2-Dichloropropane	---	---	---	---	---
cis-1,3-Dichloropropene	---	---	---	---	---
Trichloroethene	---	---	---	---	---
Dibromochloroethane	---	---	---	---	---
1,1,2-Trichloroethane	---	---	---	---	---
Benzene	---	---	---	---	---
cis-1,3-Dichloropropene	---	---	---	---	---
Trans-1,2-Dichloropropene	---	---	---	---	---
Bromoform	---	---	---	---	---
4-Methyl-2-Pentanone	---	---	---	---	---
2-Hexanone	---	---	---	---	---
Tetrachloroethene	---	---	---	---	---
1,1,2,2-Tetrachloroethane	---	---	---	---	---
Toluene	---	---	---	---	---
Chlorobenzene	---	---	---	---	---
Ethylbenzene	---	---	---	---	---
Styrene	---	---	---	---	---
Total Xylenes	---	---	---	4 J	---
SEMIVOLATILE ORGANICS					
Phenol	---	---	---	3 J	---
Benzyl Alcohol	---	---	---	---	---
1,2-Dichlorobenzene	---	---	---	---	---
4-Methylphenol	---	---	---	---	---
2,4-Dimethylphenol	---	---	---	---	---
Benzoic Acid	---	---	7 J	---	---
Naphthalene	---	---	---	---	---
4-Chloro-3-Methylphenol	---	---	---	---	---
2-Methylnaphthalene	---	---	---	---	---
Diethylphthalate	---	---	---	---	---
Di-n-Butylphthalate	2 BJ	---	2 JB	---	---
bis(2-Ethylhexyl) Phthalate	4 J	5 J	37	110	69
3-Nitroaniline	---	---	---	---	---
4-Nitroaniline	---	---	---	---	---

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TABLE B-2. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - ORGANIC RESULTS

Sample Number: MWNSL12-01	MWNSL12-02	MWNSL12-03	MWNSL12-04	DMWNSL12-04	MWNSL12-05
Sample Location: NSLMM 12	NSLMM 12	NSLMM 12	NSLMM 12	DNLSMM 12	NSLMM 12
Date Sampled: 8/24/87	8/25/87	8/26/87	8/27/87	8/27/87	8/28/87
QTR Number: EL502	EL513	EL538	EL549	EL520	EL526
VOLATILE ORGANICS: ug/l					
Chloromethane	---	---	---	---	---
Bromomethane	---	---	---	---	---
Vinyl Chloride	14 J	17 J	20 J	21 J	18 J
Chloroethane	140 J	110 J	96 J	95 J	86 J
Methylene Chloride	---	---	---	---	---
Acetone	---	---	---	7 J	---
Carbon Disulfide	---	---	---	---	---
1,1-Dichloroethene	---	---	---	---	---
1,1-Dichloroethane	10 J	12 J	13 J	12 J	11 J
1,2-Dichloroethene (Total)	9 J	10 J	10 J	10 J	9 J
Chloroform	---	---	---	---	---
1,2-Dichloroethane	---	---	---	---	---
2-Butanone	---	---	---	---	---
1,1,1-Trichloroethane	---	---	---	---	---
Carbon Tetrachloride	---	---	---	---	---
Vinyl Acetate	---	---	---	---	---
Bromodichloromethane	---	---	---	---	---
1,2-Dichloropropane	---	---	---	---	---
cis-1,3-Dichloropropene	---	---	---	---	---
Trichloroethene	---	1 J	---	---	---
Dibromochloromethane	---	---	---	---	---
1,1,2-Trichloroethane	---	---	---	---	---
Benzene	---	---	---	---	---
cis-1,3-Dichloropropene	---	---	---	---	---
Trans-1,2-Dichloropropene	---	---	---	---	---
Bromoforn	---	---	---	---	---
4-Methyl-2-Pentanone	---	4 J	---	---	---
2-Hexanone	---	---	---	---	---
Tetrachloroethene	---	---	---	---	---
1,1,2,2-Tetrachloroethane	---	---	---	---	---
Toluene	---	---	---	---	---
Chlorobenzene	---	---	---	---	---
Ethylbenzene	---	---	---	---	---
Styrene	---	---	---	---	---
Total Xylenes	---	---	---	---	---
SEMI-VOLATILE ORGANICS					
Phenol	---	---	---	---	---
Benzyl Alcohol	---	---	---	---	---
1,2-Dichlorobenzene	---	---	---	---	---
4-Methylphenol	---	---	---	---	---
2,4-Dimethylphenol	---	---	---	---	---
Benzoic Acid	---	---	---	---	7 J
Naphthalene	---	---	---	---	---
4-Chloro-3-Methylphenol	---	---	---	---	---
2-Methylnaphthalene	---	---	---	---	---
Diethylphthalate	---	---	---	---	---
Di-n-Butylphthalate	3 BJ	3 BJ	2 BJ	---	3 BJ
bis(2-Ethylhexyl) Phthalate	8 J	3 J	---	---	---
3-Nitroaniline	---	---	---	---	---
4-Nitroaniline	---	---	---	---	---

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TABLE B-2. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - ORGANIC RESULTS

Sample Number: MWNSL13-01	MWNSL13-02	DMWNSL13-02	MWNSL13-03	MWNSL13-04	MWNSL13-05
Sample Location: NSLHW 13	NSLHW 13	DMNSLHW 13	NSLHW 13	NSLHW 13	NSLHW 13
Date Sampled:	8/25/87	8/25/87	8/26/87	8/27/87	8/28/87
QTR Number:	EL511	EL532	EL537	EL547	EL525
VOLATILE ORGANICS: ug/l					
Chloroethane	NS	NR	---	---	---
Bromoethane	NS	NR	---	---	---
Vinyl Chloride	NS	NR	280 J	300 J	240 J
Chloroethane	NS	NR	150 J	---	230 J
Methylene Chloride	NS	NR	---	---	38 J
Acetone	NS	NR	---	49 J	71 J
Carbon Disulfide	NS	NR	---	---	---
1,1-Dichloroethene	NS	NR	---	---	---
1,1-Dichloroethane	NS	NR	820 J	850 J	770 J
1,2-Dichloroethene (Total)	NS	NR	610 J	680 J	620 J
Chloroform	NS	NR	---	---	---
1,2-Dichloroethane	NS	NR	18 J	17 J	---
2-Butanone	NS	NR	---	---	38 J
1,1,1-Trichloroethane	NS	NR	---	---	---
Carbon Tetrachloride	NS	NR	---	---	---
Vinyl Acetate	NS	NR	---	---	---
Bromodichloromethane	NS	NR	---	---	---
1,2-Dichloropropane	NS	NR	---	---	---
cis-1,3-Dichloropropene	NS	NR	---	---	---
Trichloroethene	NS	NR	18 J	18 J	---
Dibromochloromethane	NS	NR	---	---	---
1,1,2-Trichloroethane	NS	NR	---	---	---
Benzene	NS	NR	---	---	---
cis-1,3-Dichloropropene	NS	NR	---	---	---
Trans-1,2-Dichloropropene	NS	NR	---	---	---
Bromoform	NS	NR	---	---	---
4-Methyl-2-Pentanone	NS	NR	---	---	---
2-Hexanone	NS	NR	---	---	---
Tetrachloroethene	NS	NR	---	---	---
1,1,2,2-Tetrachloroethane	NS	NR	---	---	---
Toluene	NS	NR	---	---	---
Chlorobenzene	NS	NR	---	---	---
Ethylbenzene	NS	NR	---	---	---
Styrene	NS	NR	---	---	---
Total Xylenes	NS	NR	---	---	---
SEMIVOLATILE ORGANICS					
Phenol	NS	---	NR	---	---
Benzyl Alcohol	NS	---	NR	---	---
1,2-Dichlorobenzene	NS	---	NR	---	---
4-Methylphenol	NS	---	NR	---	---
2,4-Dimethylphenol	NS	---	NR	---	---
Benzoic Acid	NS	---	NR	---	---
Naphthalene	NS	---	NR	---	---
4-Chloro-3-Methylphenol	NS	---	NR	---	---
2-Methylnaphthalene	NS	---	NR	---	---
Diethylphthalate	NS	---	NR	---	---
Di-n-Butylphthalate	NS	2 BJ	NR	2 BJ	2 BJ
bis(2-Ethylhexyl) Phthalate	NS	---	NR	---	---
3-Nitroaniline	NS	---	NR	---	---
4-Nitroaniline	NS	---	NR	---	---

TABLE B-2. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - ORGANIC RESULTS

Sample Number: MWNSLBSA-01	DMWNSLBSA-01	MWNSLBSA-02	DMWNSLBSA-02	MWNSLBSA-03	MWNSLBSA-04	MWNSLBSA-05
Sample Location: NSLMW 8SA	DNLSLMW 8SA	NSLMW 8SA	DNLSLMW 8SA	NSLMW 8SA	NSLMW 8SA	NSLMW 8SA
Date Sampled: 8/24/87	8/24/87	8/25/87	8/25/87	8/26/87	8/27/87	8/28/87
QTR Number: EL499	EL553	EL510	EL555	EL535	EL546	EL523
=====						
VOLATILE ORGANICS: ug/l						
=====						
Chloromethane	---	---	NR	---	---	---
Bromomethane	---	---	NR	---	---	---
Vinyl Chloride	---	---	NR	---	---	---
Chloroethane	---	---	NR	---	---	---
Methylene Chloride	---	---	NR	---	---	---
Acetone	---	---	NR	---	7 J	---
Carbon Disulfide	---	---	NR	---	---	---
1,1-Dichloroethene	---	---	NR	---	---	---
1,1-Dichloroethane	---	---	NR	---	2 J	---
1,2-Dichloroethene (Total)	---	---	NR	---	---	---
Chloroform	---	---	NR	---	---	---
1,2-Dichloroethane	---	---	NR	---	---	---
2-Butanone	---	---	NR	---	---	---
1,1,1-Trichloroethane	---	---	NR	---	---	---
Carbon Tetrachloride	---	---	NR	---	---	---
Vinyl Acetate	---	---	NR	---	---	---
Bromodichloromethane	---	---	NR	---	---	---
1,2-Dichloropropane	---	---	NR	---	---	---
cis-1,3-Dichloropropene	---	---	NR	---	---	---
Trichloroethene	---	---	NR	---	---	---
Dibromochloromethane	---	---	NR	---	---	---
1,1,2-Trichloroethane	---	---	NR	---	---	---
Benzene	---	---	NR	---	---	---
cis-1,3-Dichloropropene	---	---	NR	---	---	---
Trans-1,2-Dichloropropene	---	---	NR	---	---	---
Bromofors	---	---	NR	---	---	---
4-Methyl-2-Pentanone	---	---	NR	---	---	---
2-Hexanone	---	---	NR	---	---	---
Tetrachloroethene	---	---	NR	---	---	---
1,1,2,2-Tetrachloroethane	---	---	NR	---	---	---
Toluene	---	---	NR	---	---	---
Chlorobenzene	---	---	NR	---	---	---
Ethylbenzene	---	---	NR	---	---	---
Styrene	---	---	NR	---	---	---
Total Iylenes	1 J	---	NR	---	---	---
=====						
SEMIVOLATILE ORGANICS						
=====						
Phenol	---	---	---	---	---	---
Benzyl Alcohol	---	---	---	---	---	---
1,2-Dichlorobenzene	---	---	---	---	---	---
4-Methylphenol	---	---	---	---	---	---
2,4-Dimethylphenol	---	---	---	---	---	---
Benzoic Acid	---	---	---	---	---	---
Naphthalene	---	---	---	---	---	---
4-Chloro-3-Methylpheno!	---	---	---	---	---	---
2-Methylnaphthalene	---	2 J	---	---	---	---
Diethylphthalate	---	---	---	---	---	---
Di-n-Butylphthalate	---	---	2 BJ	---	3 BJ	---
bis(2-Ethylhexyl) Phthalate	---	---	2 J	6 J	---	2 JB
3-Nitroaniline	---	---	---	---	---	---
4-Nitroaniline	---	---	---	---	---	---
=====						

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TABLE B-2. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - ORGANIC RESULTS

Sample Number: MWECC3A-01	MWECC3A-02	MWECC3A-03	MWECC3A-04	MWECC3A-05
Sample Location: ECCMW 3A	ECCMW 3A	ECCMW 3A	ECCMW 3A	ECCMW 3A
Date Sampled:	8/25/87	8/26/87	8/27/87	8/28/87
QTR Number:	EL512	EL556	EL548	EL524
VOLATILE ORGANICS: ug/l				
Chloroethane	NS	NR	---	---
Bromoethane	NS	NR	---	---
Vinyl Chloride	NS	NR	---	---
Chloroethane	NS	NR	73 J	130 J
Methylene Chloride	NS	NR	---	110 J
Acetone	NS	NR	42 J	33 J
Carbon Disulfide	NS	NR	---	32 J
1,1-Dichloroethene	NS	NR	---	---
1,1-Dichloroethane	NS	NR	63 J	79 J
1,2-Dichloroethene (Total)	NS	NR	550 J	94 J
Chloroform	NS	NR	---	650 J
1,2-Dichloroethane	NS	NR	---	750 J
2-Butanone	NS	NR	---	---
1,1,1-Trichloroethane	NS	NR	R	28 J
Carbon Tetrachloride	NS	NR	---	---
Vinyl Acetate	NS	NR	---	---
Bromodichloromethane	NS	NR	---	---
1,2-Dichloropropane	NS	NR	---	---
cis-1,3-Dichloropropene	NS	NR	---	---
Trichloroethene	NS	NR	---	---
Dibromochloromethane	NS	NR	---	---
1,1,2-Trichloroethane	NS	NR	---	---
Benzene	NS	NR	---	---
cis-1,3-Dichloropropene	NS	NR	---	---
Trans-1,2-Dichloropropene	NS	NR	---	---
Bromoform	NS	NR	---	---
4-Methyl-2-Pentanone	NS	NR	---	---
2-Hexanone	NS	NR	---	---
Tetrachloroethene	NS	NR	---	---
1,1,2,2-Tetrachloroethane	NS	NR	---	---
Toluene	NS	NR	---	---
Chlorobenzene	NS	NR	---	---
Ethylbenzene	NS	NR	---	---
Styrene	NS	NR	---	---
Total Iylenes	NS	NR	---	---
SEMIVOLATILE ORGANICS				
Phenol	NS	---	---	---
Benzyl Alcohol	NS	---	---	---
1,2-Dichlorobenzene	NS	5 J	9 J	4 J
4-Methylphenol	NS	---	---	7 J
2,4-Dimethylphenol	NS	---	---	---
Benzoic Acid	NS	---	8 J	---
Naphthalene	NS	---	---	---
4-Chloro-3-Methylphenol	NS	---	---	---
2-Methylnaphthalene	NS	---	---	---
Diethylphthalate	NS	---	---	---
Di-n-Butylphthalate	NS	2 BJ	---	---
bis(2-Ethylhexyl) Phthalate	NS	2 J	---	---
3-Nitroaniline	NS	R	R	R
4-Nitroaniline	NS	R	---	---

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TABLE B-2. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - ORGANIC RESULTS

Sample Number: MWSBP61-01	MWSBP61-02	DMWSBP61-02	MWSBP61-03	MWSBP61-04	MWSBP61-05	DMWSBP61-05
Sample Location: NSLSBP 61	NSLSBP 61	NSLDSBP 61	NSLSBP 61	NSLSBP 61	NSLSBP 61	NSLDSBP 61
Date Sampled:	8/25/87	8/25/87	8/26/87	8/27/87	8/28/87	8/28/87
QTR Number:	EL514	EL554	EL539	EL516	EL527	EL551
VOLATILE ORGANICS: ug/l						
Chloromethane	NS	NR	---	---	---	---
Bromomethane	NS	NR	---	---	---	---
Vinyl Chloride	NS	NR	---	---	---	---
Chloroethane	NS	NR	---	2 J	3 J	---
Methylene Chloride	NS	NR	---	---	---	---
Acetone	NS	NR	---	9 J	---	---
Carbon Disulfide	NS	NR	---	---	---	---
1,1-Dichloroethene	NS	NR	---	---	---	---
1,1-Dichloroethane	NS	NR	---	---	---	---
1,2-Dichloroethene (Total)	NS	NR	---	---	---	---
Chloroform	NS	NR	---	---	---	---
1,2-Dichloroethane	NS	NR	---	---	---	---
2-Butanone	NS	NR	---	5 J	---	---
1,1,1-Trichloroethane	NS	NR	---	---	---	---
Carbon Tetrachloride	NS	NR	---	---	---	---
Vinyl Acetate	NS	NR	---	---	---	---
Bromodichloromethane	NS	NR	---	---	---	---
1,2-Dichloropropane	NS	NR	---	---	---	---
cis-1,3-Dichloropropene	NS	NR	---	---	---	---
Trichloroethene	NS	NR	---	---	---	---
Dibromochloromethane	NS	NR	---	---	---	---
1,1,2-Trichloroethane	NS	NR	---	---	---	---
Benzene	NS	NR	---	---	---	---
cis-1,3-Dichloropropene	NS	NR	---	---	---	---
Trans-1,2-Dichloropropene	NS	NR	---	---	---	---
Bromofors	NS	NR	---	---	---	---
4-Methyl-2-Pentanone	NS	NR	---	---	---	---
2-Hexanone	NS	NR	---	---	---	---
Tetrachloroethene	NS	NR	---	---	---	---
1,1,2,2-Tetrachloroethane	NS	NR	---	---	---	---
Toluene	NS	NR	---	---	---	---
Chlorobenzene	NS	NR	---	---	---	---
Ethylbenzene	NS	NR	---	---	---	---
Styrene	NS	NR	---	---	---	---
Total Xylenes	NS	NR	---	---	---	---
SEMIVOLATILE ORGANICS						
Phenol	NS	NR	---	---	---	---
Benzyl Alcohol	NS	NR	---	---	---	---
1,2-Dichlorobenzene	NS	NR	---	---	---	---
4-Methylphenol	NS	NR	---	---	---	---
2,4-Dimethylphenol	NS	NR	---	---	---	---
Benzoic Acid	NS	NR	---	---	10 J	---
Naphthalene	NS	NR	---	---	---	---
4-Chloro-3-Methylphenol	NS	NR	---	---	---	---
2-Methylnaphthalene	NS	NR	---	---	---	---
Diethylphthalate	NS	NR	---	---	---	---
Di-n-Butylphthalate	NS	NR	2 BJ	3 BJ	3 BJ	---
bis(2-Ethylhexyl) Phthalate	NS	NR	---	---	---	---
3-Nitroaniline	NS	NR	---	---	---	---
4-Nitroaniline	NS	NR	---	---	---	---

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TABLE B-2. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - ORGANIC RESULTS

Sample Number: LTFB-01	LTFB-02	LTFB-03	LTFB-04	LTFB-05
Sample Location: NSLLT FB	NSLLT FB	NSLLT FB	NSLLT FB	NSLLT FB
Date Sampled:	8/25/87		8/27/87	
OTR Number:	EL534		EL522	
=====				
VOLATILE ORGANICS: ug/l				
=====				
Chloromethane	NS	---	NS	---
Bromomethane	NS	---	NS	---
Vinyl Chloride	NS	---	NS	---
Chloroethane	NS	---	NS	---
Methylene Chloride	NS	---	NS	---
Acetone	NS	---	NS	---
Carbon Disulfide	NS	---	NS	---
1,1-Dichloroethene	NS	---	NS	---
1,1-Dichloroethane	NS	---	NS	---
1,2-Dichloroethene (Total)	NS	---	NS	---
Chloroform	NS	---	NS	---
1,2-Dichloroethane	NS	---	NS	---
2-Butanone	NS	7 J	NS	R
1,1,1-Trichloroethane	NS	---	NS	---
Carbon Tetrachloride	NS	---	NS	---
Vinyl Acetate	NS	---	NS	---
Bromodichloromethane	NS	---	NS	---
1,2-Dichloropropane	NS	---	NS	---
cis-1,3-Dichloropropene	NS	---	NS	---
Trichloroethene	NS	---	NS	---
Dibromochloromethane	NS	---	NS	---
1,1,2-Trichloroethane	NS	---	NS	---
Benzene	NS	---	NS	---
cis-1,3-Dichloropropene	NS	---	NS	---
Trans-1,2-Dichloropropene	NS	---	NS	---
Bromoform	NS	---	NS	---
4-Methyl-2-Pentanone	NS	---	NS	---
2-Hexanone	NS	---	NS	---
Tetrachloroethene	NS	---	NS	---
1,1,2,2-Tetrachloroethane	NS	---	NS	---
Toluene	NS	---	NS	---
Chlorobenzene	NS	---	NS	---
Ethylbenzene	NS	---	NS	---
Styrene	NS	---	NS	---
Total Xylenes	NS	---	NS	---
=====				
SEMIVOLATILE ORGANICS				
=====				
Phenol	NS	---	NS	---
Benzyl Alcohol	NS	---	NS	---
1,2-Dichlorobenzene	NS	---	NS	---
4-Methylphenol	NS	---	NS	---
2,4-Dimethylphenol	NS	---	NS	---
Benzoic Acid	NS	---	NS	---
Naphthalene	NS	---	NS	---
4-Chloro-3-Methylphenol	NS	---	NS	---
2-Methylnaphthalene	NS	---	NS	---
Diethylphthalate	NS	---	NS	---
Di-n-Butylphthalate	NS	---	NS	2 J
bis(2-Ethylhexyl) Phthalate	NS	---	NS	---
3-Nitroaniline	NS	R	NS	R
4-Nitroaniline	NS	R	NS	---
=====				

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TABLE B-2. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - ORGANIC RESULTS

Sample Number:	MWFB-01	MWFB-02	MWFB-03	MWFB-04	MWFB-05
Sample Location:	NSLNM FB	NSLNM FB	NSLNM FB	NSLNM FB	NSLNM FB
Date Sampled:	8/24/87	8/25/87	8/26/87	8/27/87	
OTR Number:	EL508	EL533	EL544	EL521	
=====					
VOLATILE ORGANICS: ug/l					
Chloromethane	---	---	---	---	NS
Bromomethane	---	---	---	---	NS
Vinyl Chloride	---	---	---	---	NS
Chloroethane	---	---	---	---	NS
Methylene Chloride	---	---	---	---	NS
Acetone	---	---	---	---	NS
Carbon Disulfide	---	---	---	---	NS
1,1-Dichloroethene	---	---	---	---	NS
1,1-Dichloroethane	---	---	---	---	NS
1,2-Dichloroethene (Total)	---	---	---	---	NS
Chloroform	---	---	---	---	NS
1,2-Dichloroethane	---	---	---	---	NS
2-Butanone	8 J	6 J	R	R	NS
1,1,1-Trichloroethane	---	---	---	---	NS
Carbon Tetrachloride	---	---	---	---	NS
Vinyl Acetate	---	---	---	---	NS
Bromodichloromethane	---	---	---	---	NS
1,2-Dichloropropane	---	---	---	---	NS
cis-1,3-Dichloropropene	---	---	---	---	NS
Trichloroethene	---	---	---	---	NS
Dibromochloromethane	---	---	---	---	NS
1,1,2-Trichloroethane	---	---	---	---	NS
Benzene	---	---	---	---	NS
cis-1,3-Dichloropropene	---	---	---	---	NS
Trans-1,2-Dichloropropene	---	---	---	---	NS
Bromoform	---	---	---	---	NS
4-Methyl-2-Pentanone	---	---	---	---	NS
2-Hexanone	---	---	---	---	NS
Tetrachloroethene	---	---	---	---	NS
1,1,2,2-Tetrachloroethane	---	---	---	---	NS
Toluene	---	---	---	---	NS
Chlorobenzene	---	---	---	---	NS
Ethylbenzene	---	---	---	---	NS
Styrene	---	---	---	---	NS
Total Xylenes	---	---	---	---	NS
=====					
SEMI-VOLATILE ORGANICS					
Phenol	---	---	NR	---	NS
Benzyl Alcohol	---	---	NR	---	NS
1,2-Dichlorobenzene	---	---	NR	---	NS
4-Methylphenol	---	---	NR	---	NS
2,4-Dimethylphenol	---	---	NR	---	NS
Benzoic Acid	---	---	NR	---	NS
Naphthalene	---	---	NR	---	NS
4-Chloro-3-Methylphenol	---	---	NR	---	NS
2-Methylnaphthalene	---	---	NR	---	NS
Diethylphthalate	---	---	NR	---	NS
Di-n-Butylphthalate	---	---	NR	2 J	NS
bis(2-Ethylhexyl) Phthalate	---	---	NR	---	NS
3-Nitroaniline	R	R	NR	R	NS
4-Nitroaniline	R	R	NR	---	NS
=====					

TABLE B-3. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - CONVENTIONAL POLLUTANTS

Sample Number:	LT01-01	LT01-02	LT01-03	LT01-04	LT01-05	LT02-01	LT02-02	LT02-03	DLT02-03	LT02-04	LT02-05
Sample Location:	NSLLT 1	NSLLT 1	NSLLT 1	NSLLT 1	NSLLT 1	NSLLT 2	NSLLT 2	NSLLT 2	NSLLT 2	NSLLT 2	NSLLT 2
Date Sampled:	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87	8-24-87	8-25-87	8-26-87	8-26-87	8-27-87	8-28-87
Control Number:	C2607	C2615	C2626	C2640	C2665	C2605	C2614	C2627	C2630	C2641	C2666
Lab ID Number:	TC0120	TC0121	TC0142	TC0156	TC0165	TC0122	TC0123	TC0143	TC0146	TC0157	TC0166
PARAMETERS MG/L											
BOD5	34	36	39	35	36	67	68	66	68	66	67
COD	690	700	700	730	740	640	620	660	650	640	640
TOC	195.4	195.2	154.6	183.1	117.7	194.4	190.8	190.5	189.3	186.5	191.5
TSS	56	54	52	52	60	84	86	84	72	84	94
VSS	20	16	24	28	30	34	34	32	28	46	42
TDS	4030	4050	4020	3990	3970	3680	3670	3680	3650	3670	3660
TKN	220	240	240	250	250	300	300	310	300	320	310
NH3-N	230	240	240	230	230	290	290	300	300	290	290
NO3&NO2-N											
TP	0.44	0.38	0.37	0.39	0.41	0.56	0.57	0.61	0.56	0.62	0.63
ALKALINITY AS (CaCO3)	2260	2240	2240	2260	2240	2340	2360	2340	2340	2330	2350
CL	1240	1240	1240	1280	1260	1220	1220	1220	1200	1240	1240
SO4	38	28	37	26	29	33	33	32	31	32	31
DA6											
pH	NM	NM	6.9 I	7.0 II	7.0 II	NM	NM	5.7 I	NM	6.9 II	6.9 II
TEMPERATURE (degrees C)	18	22.5	27	21	19	18	22.5	28	28	21	20
CONDUCTIVITY (umhos/cm2)	4000	7000	7000	7000	7000	3650	6500	8000	8000	7000	7000

Sample Number:	LT03-01	LT03-02	LT03-03	LT03-04	LT03-05	MMNSL12-01	MMNSL12-02	MMNSL12-03	MMNSL12-04	DMMSL12-04	MMNSL12-05
Sample Location:	NSLLT 3	NSLLT 3	NSLLT 3	NSLLT 3	NSLLT 3	NSLHW 12	NSLHW 12	NSLHW 12	NSLHW 12	NSLHW 12	NSLHW 12
Date Sampled:	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87	8-24-87	8-25-87	8-26-87	8-26-87	8-27-87	8-28-87
Control Number:	C2613	C2616	C2628	C2642	C2667	C2606	C2611	C2624	C2635	C2637	C2653
Lab ID Number:	TC0124	TC0125	TC0144	TC0158	TC0167	TC0131	TC0132	TC0140	TC0151	TC0153	TC0163
PARAMETERS MG/L											
BOD5	12	11	12	17	10	33	35	31	46	34	40
COD	260	240	280	280	270	340	370	400	390	460	700
TOC	78.8	77.3	81	78.5	76.8	71.5	74.8	76.4	73.4	74.2	71.2
TSS	70	42	62	64	34	3160	3650	3900	3110	1000	8650
VSS	24	18	10	30	15	300	276	366	432	168	740
TDS	1860	1840	1820	1820	1880	2190	2160	2190	2180	2180	2220
TKN	120	120	61	130	130	58	60	64	66	70	78
NH3-N	120	12	58	120	110	55	56	57	57	58	58
NO3&NO2-N	0.2				0.1						
TP	0.41	0.3	0.42	0.35	0.18	0.5	0.82	0.95	0.82	2	5.1
ALKALINITY AS (CaCO3)	1380	1380	1320	1340	1330	670	688	812	650	650	660
CL	390	375	375	375	390	860	900	920	920	920	960
SO4	27	34	41	41	39	16	12	13	15	15	16
DA6			10								
pH	NM	NM	7.5 I	7.2 II	7.1 II	NM	NM	7.1 I	NM	7.2 II	7.2 II
TEMPERATURE (degrees C)	NM	22	27	21	20	18	23	28	28	20	18
CONDUCTIVITY (umhos/cm2)	NM	3500	3500	3300	3100	3200	2900	4000	4000	2200	3000

NOTE: NM: Not measured.

NS: Not sampled.

I: pH measured in the field.

II: pH measured in the laboratory.

--- All parameters in all samples underwent analysis. If concentration is not listed, the parameter was not detected above instrument detection limit.

TABLE B-3. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - CONVENTIONAL POLLUTANTS

Sample Number:	MWMSL13-01	MWMSL13-02	DMWMSL13-02	MWMSL13-03	DMWMSL13-03	MWMSL13-04	MWMSL13-05	MWMSL85A-01	MWMSL85A-02	MWMSL85A-03	MWMSL85A-04	MWMSL85A-05
Sample Location:	NSLHM 13	NSLHM 13	NSLHM 13	NSLHM 13	NSLHM 13	NSLHM 13	NSLHM 13	NSLHM 85A	NSLHM 85A	NSLHM 85A	NSLHM 85A	NSLHM 85A
Date Sampled:	8-25-87	8-25-87	8-25-87	8-26-87	8-26-87	8-27-87	8-28-87	8-24-87	8-25-87	8-26-87	8-27-87	8-28-87
Control Number:	C2619	C2619	C2620	C2623	C2629	C2634	C2652	C2604	C2610	C2621	C2632	C2650
Lab ID Number:	TC0126	TC0127	TC0127	TC0139	TC0145	TC0150	TC0162	TC0133	TC0134	TC0137	TC0148	TC0160
PARAMETERS MG/L												
BOD5	NS	9.3	7.3	7.3	8.8	6.7	12	1.6	2.5	2.5	5.5	2.1
COD	NS	57	57	58	84	49	100	15	7	15	7	12
TOC	NS	15.5	14.7	15.7	16.2	15.8	18.2	2.8	2.4	2.8	2.6	2.5
TSS	NS	16	16	22	24	24	168	42	4	123	105	92
VSS	NS	4	6	4	6	6	28	8	2	20	15	15
TDS	NS	826	837	790	810	778	764	334	338	328	328	406
TKN	NS	16	16	21	18	16	18	1.2	1.1	1.3	1.2	1.2
NH3-N	NS	16	16	16	16	15	15	1	1	1	1	1
NO3&NO2-N	NS	---	---	---	---	---	---	---	---	---	---	---
TP	NS	---	---	---	---	---	0.24	0.11	0.03	0.11	0.05	0.07
ALKALINITY AS (CaCO3)	NS	514	520	504	504	500	480	322	326	330	328	328
CL	NS	160	160	150	150	145	140	5	4	5	5	5
SO4	NS	36	34	34	34	36	38	6	6	6	7	10
DB6	NS	---	---	---	---	---	---	---	---	---	---	---
pH	NS	NM	NM	7.0	NM	6.9	7	NM	NM	6.3	7.3	7.4
TEMPERATURE (degrees C)	NS	22	22	27	27	21	18	18	23	28	21	22
CONDUCTIVITY (uamhos/cm2)	NS	1300	1300	1400	1400	1300	1100	800	550	700	600	490

Sample Number:	MNECC3A-01	MNECC3A-02	MNECC3A-03	MNECC3A-04	MNECC3A-05	MWSBP61-01	MWSBP61-02	MWSBP61-03	MWSBP61-04	MWSBP61-05	DMWSBP61-05
Sample Location:	ECCHM 3A	ECCHM 3A	ECCHM 3A	ECCHM 3A	ECCHM 3A	NSLSBP 61	NSLSBP 61	NSLSBP 61	NSLSBP 61	NSLSBP 61	NSLSBP 61
Date Sampled:	8-25-87	8-25-87	8-26-87	8-27-87	8-28-87	8-25-87	8-26-87	8-26-87	8-27-87	8-28-87	8-28-87
Control Number:	C2617	C2617	C2622	C2633	C2651	C2612	C2612	C2625	C2636	C2664	C2668
Lab ID Number:	TC0128	TC0128	TC0138	TC0149	TC0161	TC0136	TC0136	TC0141	TC0152	TC0164	TC0168
PARAMETERS MG/L											
BOD5	NS	24	27	21	27	NS	29	31	23	25	27
COD	NS	350	340	360	370	NS	630	440	520	370	350
TOC	NS	77.2	77.2	75.2	78.8	NS	91	96	97	98.6	97
TSS	NS	2760	1310	840	2360	NS	9640	4470	5920	1520	1150
VSS	NS	228	172	138	344	NS	576	308	420	184	148
TDS	NS	2340	2260	2280	2410	NS	2630	2710	2790	2800	2830
TKN	NS	63	54	65	72	NS	74	77	76	93	90
NH3-N	NS	59	57	58	58	NS	57	63	66	69	68
NO3&NO2-N	NS	---	---	---	---	NS	---	---	---	---	---
TP	NS	1.2	0.7	1.2	1.3	NS	5.8	2.4	4	0.72	0.6
ALKALINITY AS (CaCO3)	NS	700	732	710	730	NS	1240	1300	1340	1380	1360
CL	NS	940	920	940	940	NS	880	920	920	960	940
SO4	NS	15	13	14	15	NS	14	24	17	15	28
DB6	NS	---	---	---	---	NS	---	---	---	---	---
pH	NS	NM	6.9	7.0	7.1	NS	NM	6.7	6.7	6.7	6.7
TEMPERATURE (degrees C)	NS	23	27	19	17	NS	22	25	20	18	NM
CONDUCTIVITY (uamhos/cm2)	NS	3100	4000	3400	3400	NS	3500	4600	4200	4000	NM

NOTE: NM: Not measured.

NS: Not sampled.

! : pH measured in the field.

!! : pH measured in the laboratory.

*---- All parameters in all samples underwent analysis. If concentration is not listed, the parameter was not detected above instrument detection limit.

TABLE B-3. NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
LEACHATE AND GROUNDWATER - CONVENTIONAL POLLUTANTS

Sample Number: LTFB-01 Sample Location: NSLLT FB Date Sampled: 8-24-87 Control Number: C2608 Lab ID Number: TC0135	LTFB-02 NSLLT FB	LTFB-03 NSLLT FB	LTFB-04 NSLLT FB 8-27-87 C2639 TC0155	LTFB-05 NSLLT FB	MWFB-01 NSLNM FB 8-24-87 C2609 TC0129	MWFB-02 NSLNM FB 8-25-87 C2610 TC0130	MWFB-03 NSLNM FB 8-26-87 C2631 TC0147	MWFB-04 NSLNM FB 8-27-87 C2638 TC0154	MWFB-05 NSLNM FB
PARAMETERS MG/L									
BOD5	---	NS	NS	---	NS	---	---	---	NS
COD	---	NS	NS	---	NS	---	---	---	NS
TDC	---	NS	NS	---	NS	---	---	---	NS
TSS	---	NS	NS	---	NS	---	---	---	NS
VSS	1	NS	NS	---	NS	---	1	1	NS
TDS	---	NS	NS	---	NS	---	---	---	NS
TKN	---	NS	NS	---	NS	---	---	---	NS
NH3-N	---	NS	NS	---	NS	---	---	---	NS
NO3&NO2-N	---	NS	NS	---	NS	---	---	---	NS
TP	---	NS	NS	---	NS	---	---	---	NS
ALKALINITY AS (CaCO3)	2	NS	NS	2	NS	2	2	2	NS
CL	---	NS	NS	---	NS	---	---	---	NS
SO4	---	NS	NS	---	NS	---	---	---	NS
ORP	---	NS	NS	---	NS	---	---	---	NS
pH	NM	NS	NS	8.5 ^{ff}	NS	NM	NM	NM	8.5 ^{ff}
TEMPERATURE (degrees C)	NM	NS	NS	NM	NS	NM	NM	NM	NM
CONDUCTIVITY (uohms/cm2)	NM	NS	NS	NM	NS	NM	NM	NM	NM

NOTE: NM: Not measured.

NS: Not sampled.

8: pH measured in the field.

ff: pH measured in the laboratory.

----- All parameters in all samples underwent analysis. If concentration is not listed, the parameter was not detected above instrument detection limit.

TABLE D-1
NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
GROUNDWATER - METALS

UNFILTERED												
Sample Number:	ECCDN01	ECCNM1A	ECCNM12	ECCNM13	ECCNM13D	ECCNM14	ECCNM14D	ECCNM15	ECCNM16	ECCNM17	ECCNM18	ECCNM19A
Sample Location:		ECCNM1A	ECCNM12	ECCNM13	ECCNM13	ECCNM14	ECCNM14	ECCNM15	ECCNM16	ECCNM17	ECCNM18	ECCNM19A
ITR Number:	MET356	MET325	MET373	MET363	MET365	MET329	MET331	MET333	MET335	MET337	MET339	MET367
Date Sampled:	04/25/88	04/27/88	04/28/88	04/28/88	04/28/88	04/28/88	04/28/88	04/27/88	04/27/88	04/27/88	04/27/88	04/28/88
Sample Type:	DRILLERS H2O	GRAB	GRAB	GRAB	DUPL	GRAB	DUPL	GRAB	GRAB	GRAB	GRAB	GRAB
INORGANICS (ug/l)												
Aluminum	559 J	68 J,B	10300 J	138 J,B	13800 J	4440 J	3520 J	9200 J	14300 J	21100 J	9420 J	7630 J
Antimony	---	---	---	---	---	---	---	---	---	---	---	---
Arsenic	---	---	18 J	---	13 J	2.6 J,B	4 J,B	11 J	5 J	28 J	11 J	28 J
Barium	52 J	313	253	337	467	422	461	513	609	1140	578	657
Beryllium	---	---	---	---	---	---	---	---	---	2.5 J	---	---
Cadmium	---	---	4.8 J	---	5.5 J	5.1 J	3.5 J	5 J	5.9 J	14 J	6.8 J	5.3 J
Calcium	60800	120000	238000	314000	431000	186000	240000	222000	321000	516000	305000	320000
Chromium	5 J,B	7 J,B	33	---	28	18 B	15 B	26	34	50	23 B	27
Cobalt	---	---	18 J	8 J	41 J	11 J	10 J	12 J	25 J	50 J	16 J	16 J
Copper	17 J,B	3 J,B	42 J,B	---	169 J	45 J,B	66 J,B	114 J	227 J	423 J	64 J	40 J,B
Iron	3560 J	2680 J	25300	5560	49000	24600 J	28600	42600 J	44300 J	88900 J	45700 J	27400
Lead	6 J	3.9 J	48 J	3.6 J,B	115 J	31 J,B	47 J	74 J	214 J	723 J	33 J	33 J
Magnesium	16500	32900	44870	43100	88500	54900	62300	60900	74700	140000	74400	91100
Manganese	91 J	55 J	917 J	1080 J	1760 J	559 J	790 J	781 J	909 J	2600 J	1020 J	1060 J
Mercury	---	---	---	0.4	0.3	---	---	---	0.2 J	0.9 J	---	---
Nickel	---	---	60 J	21 J	100 J	30 J	34 J	42 J	78 J	135 J	49 J	42 J
Potassium	2670 J	1480 J	18700	1800 J	3290 J	2400 J	2360 J	2910 J	5460	5960	3270 J	2640 J
Selenium	---	---	---	---	7 R	---	---	---	25 J	147 J	---	---
Silver	---	---	---	---	---	---	---	---	---	---	---	---
Sodium	12500	9340	15300	24400	24600	15800	16200	15200	24600	16200	13600	21600
Thallium	---	---	---	---	---	---	---	---	---	---	---	---
Vanadium	---	---	25 J	---	26 J	15 J	12 J	25 J	38 J	45 J	19 J	29 J
Zinc	38 J	5 J	113 J	14 J	279 J	255 J	123 J	195 J	295 J	416 J	143 J	86 J

Sample Number:	ECCNM19B	ECCNM20	ECCNM21	ECCNM22	ECCNM22D	ECCNM23	ECCSUMP01	ECCNM96	ECCNM98	ECCNM99
Sample Location:	ECCNM19B	ECCNM20	ECCNM21	ECCNM22	ECCNM22	ECCNM23	SUMP			
ITR Number:	MET369	MET342	MET327	MET379	MET381	MET383	MET371	MET385	MET375	MET341
Date Sampled:	04/28/88	04/27/88	04/27/88	05/3/88	05/3/88	05/3/88	04/28/88	05/3/88	04/28/88	04/27/88
Sample Type:	GRAB	GRAB	GRAB	GRAB	DUPL	GRAB	GRAB	FIELD BLANK	FIELD BLANK	FIELD BLANK
INORGANICS (ug/l)										
Aluminum	171 J	27900 J	14300 J	280000 J	277000 J	221000 J	235 J	26.5 J,B	28 J,B	29 J,B
Antimony	---	---	---	---	---	---	---	---	13 J,B	52 J,B
Arsenic	---	32 J	17 J	2.2 J	---	2.1 J	9 J,B	---	2.5 J	---
Barium	392	1050	660	2430 J	2380 J	2030 J	48 J	3.7 J,B	---	---
Beryllium	---	2.6 J	---	10.8 J	12.6 J	9.9 J	---	---	---	---
Cadmium	---	13	7.5	---	7.9 J	36.9 J	---	---	---	---
Calcium	81200	514000	514000	2439000	3365000	2003000	57600	56.6 J,B	449 J	741 J
Chromium	9 J,B	65	25	233	238	286	6 J,B	---	5 J	5 J
Cobalt	---	52	25 J	245	282	194	---	---	---	---
Copper	5 J,B	350 J	113 J	1380 J	1660 J	1220 J	4 J,B	5.1 J,B	15 J	12 J
Iron	2950	92900 J	76000 J	825000 J	944000 J	468000 J	8550	15.6 J,B	---	64 R
Lead	---	278 J	122 J	15.1 J	2.9 J,B	2.8 J,B	7.7 B	2.2 J	6.3 J	---
Magnesium	27400	130000	103000	738000	995000	592000	5710	---	23	147 J
Manganese	79 J	2330 J	2220 J	14500 J	17900 J	9460 J	203 J	---	---	3 J
Mercury	---	0.4 J	0.2 J	---	---	---	---	---	---	---
Nickel	7 J	165 J	76 J	599 J	655 J	589 J	6 J	---	---	---
Potassium	1410 J	11500	4440 J	40000	40200	37800	8810	---	---	283 J
Selenium	---	---	---	---	19.5 J	34.9 J	---	---	---	---
Silver	---	---	---	3.1 J,B	---	---	---	---	---	---
Sodium	13200	57000	13300	51300	53200	77300	6980	120 J,B	131 J	224 J
Thallium	---	---	---	---	---	---	---	---	---	---
Vanadium	5 J	66 J	22 J	541 J	596 J	547 J	---	---	---	---
Zinc	3 J	534 J	324 J	2430 J	2770 J	2050 J	5 J	3.7 J,B	---	---

--- All compounds in all samples underwent analysis. If concentration is not listed, the compound was not detected above instrument detection limit.

J Estimated value; or the report value is less than the contract required detection limit but greater than the instrument detection limit.

B Compound present at equal to or less than five times the concentration present in the laboratory or field blank.

R Data not usable, indicates possible false negative, or very low spike sample recovery.

TABLE D-1

NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
GROUNDWATER - METALS

FILTERED												
Sample Number:	ECCNW01(F)	ECCNW10(F)	ECCNW12(F)	ECCNW13(F)	ECCNW13D(F)	ECCNW14(F)	ECCNW14D(F)	ECCNW15(F)	ECCNW16(F)	ECCNW17(F)	ECCNW18(F)	ECCNW19A(F)
Sample Location:	ECCNW01	ECCNW10	ECCNW12	ECCNW13	ECCNW13	ECCNW14	ECCNW14	ECCNW15	ECCNW16	ECCNW17	ECCNW18	ECCNW19A
ITR Number:	MET359	MET326	MET374	MET364	MET366	MET330	MET332	MET334	MET336	MET338	MET340	MET368
Date Sampled:	04/25/88	04/27/88	04/28/88	04/28/88	04/28/88	04/28/88	04/28/88	04/27/88	04/27/88	04/27/88	04/27/88	04/28/88
Sample Type:	DRILLERS H2O	GRAB	GRAB	GRAB	DUPL	GRAB	DUPL	GRAB	GRAB	GRAB	GRAB	GRAB
INORGANICS (ug/l)												
Aluminum	18 J,B	77 J,B	55 J,B	21 J,B	168 J,B	48 J,B	28 J,B	31 J,B	36 J,B	123 J,B	382 J	534 J
Antimony	---	---	---	---	---	21 J,B	---	---	---	---	---	---
Arsenic	---	---	12	16 J	19 J	---	---	---	---	2.1 J	---	24 J
Barium	64 J	316 B	161 J,B	279 B	290 B	371 B	356 B	348 B	382 B	508	540	404 B
Beryllium	---	---	---	---	---	---	---	---	---	---	---	---
Cadmium	---	3 J,B	---	---	---	2.7 J	---	2.1 J	---	---	3.1 J,B	---
Calcium	52500	86200	108000	157000	162000	89800	89200	90400	147000	96600	179000	92600
Chromium	---	8 J,B	7 J,B	5 J,B	8 J,B	8 J,B	6 J,B	11 J,B	8 J,B	8 J,B	10 J,B	12 J,B
Cobalt	---	---	---	---	7 J	---	---	---	---	---	---	---
Copper	6 J	15 J,B	4 J,B	3 J,B	5 J,B	3 J,B	5 J,B	5 J,B	---	4 J,B	8 J,B	9 J,B
Iron	---	1990 J	621 J	12500 J	10300 J	570 J	178 J,B	516 J	4420 J	408 J,B	6220 J	5220 J
Lead	---	10 J	---	7.3 J	7.3 J	19 J	19 J	3.9 J	3.3 J	---	11 J	4 J
Magnesium	14800	31100	13000	41300	42500	33400	33600	34200	39400	34100	37000	31200
Manganese	46	51	225	684	746	37	33	45	263	97	402	436
Mercury	---	0.2 J	---	---	---	---	---	---	---	0.2 J	0.2 J	---
Nickel	---	6 J	16 J	16 J	19 J	9 J	8 J	6 J	11 J	8 J	6 J	11 J
Potassium	2480 J	1380 J,B	16500	1740 J	2060 J	1560 J	1440 J	1180 J,B	3400 J	1920 J	1460 J,B	1780 J
Selenium	---	---	---	---	---	---	---	---	---	---	---	---
Silver	---	---	---	---	---	---	---	---	---	---	---	---
Sodium	11700	9640	16000	24300	26000	16200	15900	14800	24400	16300	13800	21100
Thallium	---	---	---	---	---	---	---	---	---	---	---	---
Vanadium	---	---	---	---	---	5 J	4 J	---	---	---	---	6 J
Zinc	38 J	305 J	177 J,B	106 J,B	77 J,B	85 J,B	12 J,B	38 J,B	56 J,B	100 J,B	126 J,B	213 J
INORGANICS (ug/l)												
Sample Number:	ECCNW19B(F)	ECCNW20(F)	ECCNW21(F)	ECCNW22(F)	ECCNW22D(F)	ECCNW23(F)	ECCSUMP01(F)	ECCNW96(F)	ECCNW98(F)	ECCNW99(F)		
Sample Location:	ECCNW19B	ECCNW20	ECCNW21	ECCNW22	ECCNW22	ECCNW23(F)	SUMP	ECCNW96	ECCNW98	ECCNW99		
ITR Number:	MET370	MET343	MET328	MET380	MET382	MET384	MET372	MET386	MET376	MET344		
Date Sampled:	04/28/88	04/27/88	04/27/88	05/3/88	05/3/88	05/3/88	04/28/88	05/3/88	04/28/88	04/27/88		
Sample Type:	GRAB	GRAB	GRAB	GRAB	DUPL	GRAB	GRAB	FIELD BLANK	FIELD BLANK	FIELD BLANK		
Aluminum	43 J,B	26 J,B	254 J	1970 J	173 J,B	81.4 J,B	36 J,B	72.8 J,B	37 J,B	47 J,B		
Antimony	---	15 J,B	---	---	---	---	---	---	---	72 B		
Arsenic	2 J	4 J	---	6.9 J	7.9 J	3.8 J	7 J	---	---	---		
Barium	404 B	570	320 B	398 J,B	381 J,B	455 J,B	109 J,B	106 J	100 J	81 J		
Beryllium	---	---	---	---	---	---	---	---	---	---		
Cadmium	---	1.9 J,B	---	---	---	---	---	---	---	2.8 J		
Calcium	80500	102000	104000	102000	93500	145000	53400	268 J,B	480 J	1120 J		
Chromium	9 J,B	11 J,B	6 J,B	12.3 J,B	7.4 J,B	12.5 J,B	6 J,B	9.4 J	5 J	7 J		
Cobalt	---	---	---	6.5 J,B	---	---	---	5.9 J	---	---		
Copper	4 J,B	3 J,B	4 J,B	21.2 J,B	8.3 J,B	8.9 J,B	3 J,B	9.6 J,B	10 J	10 J		
Iron	2110 J	82 J	1440 J	5140 J	348 J,B	172 J,B	4680 J	97.5 J,B	37 J	85 R		
Lead	---	---	4.8 J	16.5 J	4 J,B	3.8 J,B	---	2.2 J	---	---		
Magnesium	27400	42900	30300	35100	32400	48700	5250	---	52 J	171 J		
Manganese	69	200	142	196	128	231	181	---	---	4 J		
Mercury	---	---	---	---	---	---	---	---	---	---		
Nickel	---	13 J	---	24.8 J,B	18.2 J,B	23.4 J,B	6 J	18.6 J,B	---	---		
Potassium	1480 J	7060	1280 J,B	2970 J	2770 J	3940 J	8330	---	209 J	320 J		
Selenium	---	---	---	---	---	---	---	---	---	---		
Silver	---	---	---	---	---	---	---	---	---	---		
Sodium	14000	56400	12800	51400	49900	69900	7100	442 J,B	573 J	733 J		
Thallium	---	---	---	---	---	---	---	---	---	---		
Vanadium	---	4 J	---	---	---	---	---	---	---	---		
Zinc	52 J,B	25 J,B	56 J,B	144 J,B	95.5 J,B	123 J,B	95 J,B	37.7 J,B	38 J	55 J		

--- All compounds in all samples underwent analysis. If concentration is not listed, the compound was not detected above instrument detection limit.
 J Estimated value; or the report value is less than the contract required detection limit but greater than the instrument detection limit.
 B Compound present at equal to or less than five times the concentration present in the laboratory or field blank.

low spike sample recovery

TABLE D-2

NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
GROUNDWATER - ORGANIC RESULTS

Sample Number:	ECCDW01	ECCMW1A	ECCMW12	ECCMW13	ECCMW13	ECCMW13B	ECCMW14	ECCMW14D	ECCMW15	ECCMW16	ECCMW17	ECCMW18	ECCMW19A
Sample Location:	ECCMW1A	ECCMW1A	ECCMW12	ECCMW13	ECCMW13	ECCMW13	ECCMW14	ECCMW14	ECCMW15	ECCMW16	ECCMW17	ECCMW18	ECCMW19A
OTR Number:	ES728	EW781	EW800	EW794	EW796	EW795	EW783	EW784	EW785	EW786	EW787	EW788	EW797
Date Sampled:	04/25/88	04/27/88	04/28/88	04/28/88	04/28/88	04/28/88	04/28/88	04/28/88	04/27/88	04/27/88	04/27/88	04/27/88	04/28/88
Sample type:	DRILLER H2O	GRAB	GRAB	GRAB	MSD	DUPL	GRAB	DUPL	GRAB	GRAB	GRAB	GRAB	GRAB

VOLATILE ORGANICS (ug/l)

Chloroethane	--	--	--	--	--	--	--	--	--	--	--	--	--
Bromoethane	--	--	--	--	--	--	--	--	--	--	--	--	--
Vinyl Chloride	--	--	--	2800	3600	3800	--	--	--	--	--	--	--
Chloroethane	--	--	--	--	--	--	--	--	--	--	--	--	--
Methylene Chloride	--	--	12000	109 J	--	--	--	--	--	--	--	--	--
Acetone	--	--	11000	--	--	--	--	--	--	210	--	--	--
Carbon Disulfide	--	--	--	--	--	--	--	--	--	--	--	--	--
1,1-Dichloroethene	--	--	--	--	--	--	--	--	--	--	--	--	--
1,1-Dichloroethane	--	--	3700	--	75 J	79 J	--	--	--	15	--	--	--
1,2-Dichloroethene (Total)	--	--	72000	--	--	--	--	--	--	--	--	--	--
Chloroform	71	--	5300	--	--	--	--	--	--	--	--	--	35000
1,2-Dichloroethane	--	--	--	--	--	--	--	--	--	--	--	--	--
2-Butanone	--	--	R	R	R	R	R	R	--	--	--	--	R
1,1,1-Trichloroethane	24	--	64000	--	--	--	--	--	--	--	--	--	21000
Carbon Tetrachloride	--	--	--	--	--	--	--	--	--	--	--	--	--
Vinyl Acetate	--	--	--	--	--	--	--	--	--	--	--	--	--
Bromodichloromethane	6	--	--	--	--	--	--	--	--	--	--	--	--
1,2-Dichloropropane	--	--	--	--	--	--	--	--	--	--	--	--	--
cis-1,3-Dichloropropene	--	--	--	--	--	--	--	--	--	--	--	--	--
Trichloroethene	36	--	16000	--	--	2 J	--	--	--	--	--	--	11000
Dibromochloromethane	--	--	--	--	--	--	--	--	--	--	--	--	--
1,1,2-Trichloroethane	--	--	--	--	--	--	--	--	--	--	--	--	--
Benzene	--	--	--	--	--	--	--	--	--	4 J	--	--	--
cis-1,3-Dichloropropene	--	--	--	--	--	--	--	--	--	--	--	--	--
Trans-1,2-Dichloropropene	--	--	--	--	--	--	--	--	--	--	--	--	--
Bromofors	--	--	--	--	--	--	--	--	--	--	--	--	--
4-Methyl-2-Pentanone	--	--	--	--	--	--	--	--	--	--	--	--	--
2-Hexanone	--	--	--	--	--	--	--	--	--	--	--	--	--
Tetrachloroethene	28	--	13000	--	--	--	--	--	--	--	--	--	1200 J
1,1,2,2-Tetrachloroethane	--	--	--	--	--	--	--	--	--	--	--	--	--
Toluene	1 J	--	7200 J	93 J	130 J	140 J	--	--	--	0.9 J	--	--	--
Chlorobenzene	--	--	--	--	--	--	--	--	--	--	--	--	--
Ethylbenzene	--	--	--	--	--	--	--	--	--	--	--	--	850 J
Styrene	--	--	--	--	--	--	--	--	--	--	--	--	--
Total Tylenes	--	--	--	--	--	--	--	--	--	--	--	--	2000 J

SEMI-VOLATILE ORGANICS

Phenol	--	--	--	--	--	--	--	--	--	--	--	--	--
1,2-Dichlorobenzene	--	--	67	--	--	--	--	--	--	--	--	--	310
1,4-Dichlorobenzene	--	--	--	--	--	--	--	--	--	--	--	--	65
2-Methylphenol	--	--	--	--	21 J	--	--	--	--	--	--	--	--
4-Methylphenol	--	--	290	--	--	--	--	--	--	--	--	--	--
2,4-Dimethylphenol	--	--	300	10 J	10 J	5 J	--	--	--	--	--	--	--
Isophorone	--	--	120	--	--	--	--	--	--	--	--	--	--
Naphthalene	--	--	28	--	--	--	--	--	--	--	--	--	--
2-Methylnaphthalene	--	--	--	--	--	--	--	--	--	--	--	--	6 J
Diethylphthalate	--	--	130	19 J	4 J	4 J	--	--	6 J	--	--	--	--
Di-n-Butylphthalate	4 J	5 J	3 J	--	3 J	3 J	--	--	--	4 J	4 J	--	--
bis(2-Ethylhexyl) Phthalate	22 B	15 JB	49 B	26 B	27 B	28 B	16 JB	37 JB	21 B	18 JB	13 JB	13 JB	15 JB
Dimethyl Phthalate	--	--	57	--	--	--	--	--	--	--	--	--	--
2-Nitroaniline	--	--	-- R	-- R	-- R	-- R	-- R	-- R	--	--	--	--	-- R
3-Nitroaniline	--	--	-- R	-- R	-- R	-- R	-- R	-- R	--	--	--	--	-- R
4-Nitroaniline	-- R	-- R	-- R	-- R	-- R	-- R	-- R	-- R	-- R	-- R	-- R	-- R	-- R
4,6-Dinitro-2-Methylphenol	--	--	-- R	-- R	-- R	-- R	-- R	-- R	--	--	--	--	-- R
N-Nitrosodiphenylamine	--	--	-- R	-- R	-- R	-- R	-- R	-- R	--	--	--	--	-- R

--- All compounds in all samples underwent analysis. If concentration is not listed, the compound was not detected above quantification detection limits.

J Estimated value; or the report value is less than the contract required detection limit but greater than the quantification detection limit.

B Compound present at equal to or less than five times the concentration present in the field blank.

N/A Not analyzed.

R Unusable, indicates possible false negative.

TABLE D-2

NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
GROUNDWATER - ORGANIC RESULTS

Sample Number: ECCMW196	ECCMW20	ECCMW21	ECCMW22	ECCMW22D	ECCMW23	ECCSUMP01	ECCMW96	ECCMW97	ECCMW98	ECCMW99
Sample Location: ECCMW196	ECCMW20	ECCMW21	ECCMW22	ECCMW22D	ECCMW23	SUMP				
DR Number: EW798	EW789	EW782	ES733	ES734	ES735	EW799	ES736	EW732	ES729	EW790
Date Sampled: 04/28/88	04/27/88	04/27/88	05/03/88	05/03/88	05/03/88	04/28/88	05/03/88	04/28/88	04/28/88	04/27/88
Sample type: GRAE	GRAE	GRAE	GRAE	DUPL	GRAE	GRAE	FIELD BLANK	TRIP BLANK	FIELD BLANK	FIELD BLANK
VOLATILE ORGANICS (ug/l)										
Chloromethane	--	--	--	--	--	--	--	--	--	--
Bromomethane	--	--	--	--	--	--	--	--	--	--
Vinyl Chloride	--	100	--	--	--	--	--	--	--	--
Chloroethane	--	27	--	--	--	--	--	--	--	--
Methylene Chloride	--	--	--	--	--	3200	--	--	--	--
Acetone	--	--	--	--	--	--	--	--	--	--
Carbon Disulfide	--	--	--	--	--	--	--	--	--	--
1,1-Dichloroethene	--	--	--	--	--	--	--	--	--	--
1,1-Dichloroethane	220	26	--	--	--	1800	--	--	--	--
1,2-Dichloroethene (Total)	330	230	--	--	--	16000	--	--	--	--
Chloroform	--	--	--	--	--	850 J	--	--	--	8
1,2-Dichloroethane	--	--	--	--	--	--	--	--	--	--
2-Butanone	-- R	--	--	--	--	-- R	--	-- R	-- R	--
1,1,1-Trichloroethane	360	5	--	--	--	13000	--	--	--	--
Carbon Tetrachloride	--	--	--	--	--	--	--	--	--	--
Vinyl Acetate	--	--	--	--	--	--	--	--	--	--
Bromodichloromethane	--	--	--	--	--	--	--	--	--	--
1,2-Dichloropropane	--	--	--	--	--	--	--	--	--	--
cis-1,3-Dichloropropene	--	--	--	--	--	--	--	--	--	--
Trichloroethene	1700	--	--	--	--	1300	--	--	--	--
Dibromochloromethane	--	--	--	--	--	--	--	--	--	--
1,1,2-Trichloroethane	--	--	--	--	--	--	--	--	--	--
Benzene	--	--	--	--	--	--	--	--	--	--
cis-1,3-Dichloropropene	--	--	--	--	--	--	--	--	--	--
Trans-1,2-Dichloropropene	--	--	--	--	--	--	--	--	--	--
Bromocloro	--	--	--	--	--	--	--	--	--	--
4-Methyl-2-Pentanone	--	--	--	--	--	--	--	--	--	--
2-Hexanone	--	--	--	--	--	--	--	--	--	--
Tetrachloroethene	110	--	--	--	--	430 J	--	--	--	--
1,1,2,2-Tetrachloroethane	--	--	--	--	--	--	--	--	--	--
Toluene	--	--	--	--	--	700 J	--	--	--	--
Chlorobenzene	--	--	--	--	--	--	--	--	--	--
Ethylbenzene	--	--	--	--	--	--	--	--	--	--
Styrene	--	--	--	--	--	--	--	--	--	--
Total Xylenes	--	--	--	--	--	--	--	--	--	--
SEMI-VOLATILE ORGANICS										
Phenol	--	--	--	6 JB	6 JB	13 J	7 J	N/A	--	--
1,2-Dichlorobenzene	66	--	--	--	--	85	--	N/A	--	--
1,4-Dichlorobenzene	--	--	--	--	--	--	--	N/A	--	--
2-Methylphenol	--	--	--	--	--	140	--	N/A	--	--
4-Methylphenol	--	--	--	--	--	35	--	N/A	--	--
2,4-Dimethylphenol	--	--	--	--	--	51	--	N/A	--	--
Isophorone	--	--	--	--	--	35	--	N/A	--	--
Naphthalene	--	--	--	--	--	5 J	--	N/A	--	--
2-Methylnaphthalene	--	--	--	--	--	--	--	N/A	--	--
Diethylphthalate	--	--	--	--	--	32	--	N/A	--	--
Di-n-Butylphthalate	--	--	5 J	--	--	4 J	--	N/A	--	--
bis(2-Ethylhexyl) Phthalate	34 B	8 JB	26 B	5 J	--	36 B	--	N/A	25	15 J
Dimethyl Phthalate	--	--	--	--	--	8 J	--	N/A	--	--
2-Nitroaniline	-- R	--	--	--	--	-- R	--	N/A	-- R	--
3-Nitroaniline	-- R	--	--	--	--	-- R	--	N/A	-- R	--
4-Nitroaniline	-- R	-- R	-- R	--	--	-- R	--	N/A	-- R	-- R
4,6-Dinitro-2-Methylphenol	--	--	--	--	--	--	--	N/A	--	--
N-Nitrosodiphenylamine	-- R	--	--	--	--	-- R	--	N/A	-- R	--

--- All compounds in all samples underwent analysis. If concentration is not listed, the compound was not detected above quantification detection limits.
 J Estimated value; or the report value is less than the contract required detection limit but greater than the quantification detection limit.
 R Compound present at equal to or less than five times the concentration present in the field blank.
 N/A Not analyzed.
 R Unusable, indicates possible false negative.

TABLE D-3

NSL/ECC PREDESIGN INVESTIGATION ANALYTICAL RESULTS
GROUNDWATER - CONVENTIONAL PARAMETERS

Sample Number:	ECCMW1A	ECCMW12	ECCMW13	ECCMW13D	ECCMW14	ECCMW14D	ECCMW15	ECCMW16	ECCMW17	ECCMW18	ECCMW19A
Sample Location:	ECCMW1A	ECCMW12	ECCMW13	ECCMW13	ECCMW14	ECCMW14	ECCMW15	ECCMW16	ECCMW17	ECCMW18	ECCMW19A
Control Number:	A2840	A2854	A2850	A2851	A2848	A2849	A2842	A2843	A2844	A2845	A2852
Date Sampled:	04/27/88	04/28/88	04/28/88	04/28/88	04/28/88	04/28/88	04/27/88	04/27/88	04/27/88	04/27/88	04/28/88
Sample type:	GRAB	GRAB	GRAB	DUPL	GRAB	DUPL	GRAB	GRAB	GRAB	GRAB	GRAB
CONVENTIONAL PARAMETERS (mg/L)											
COD	---	510	340	280	120	120	160	210	720	160	140
TDS	418	647	795	790	453	454	460	750	477	428	522
TSS	78 J	1440 J	4750 J	4580 J	1400 J	1230 J	1530 J	2530 J	8480 J	1960 J	2160 J
ALKALINITY (as CaCO ₃)	313	255	459	460	495	458	552	686	1052	552	376
NH ₃ -N	0.2	5.6	1.1	1.2	0.6	0.6	0.6	1.0	0.9	0.6	1.0
CHLORIDES	12	75	81	78	21	21	21	68	24	13	47
Sample Number:	ECCMW19B	ECCMW20	ECCMW21	ECCMW22	ECCMW22D	ECCMW23	ECCSUMP01	ECCMW96	ECCMW98	ECCMW99	
Sample Location:	ECCMW19B	ECCMW20	ECCMW21	ECCMW22	ECCMW22	ECCMW23	SUMP				
Control Number:	A2853	A2846	A2841	A2858	A2859	A2860	A2855	A2861	A2856	A2847	
Date Sampled:	04/28/87	04/27/88	04/27/88	05/03/88	05/03/88	05/03/88	04/28/88	05/03/88	04/28/88	04/27/88	
Sample type:	GRAB	GRAB	GRAB	GRAB	DUPL	GRAB	GRAB	FIELD BLANK	FIELD BLANK	FIELD BLANK	
CONVENTIONAL PARAMETERS (mg/L)											
COD	---	580	300	1800	1400	1700	130	10	8	---	
TDS	406	773	431	598	605	973	647	---	13	6	
TSS	27 J	7290 J	4610 J	28940 J	18720 J	16900 J	32 J	---	---	---	
ALKALINITY (as CaCO ₃)	341	1004	731	1507	1511	1008	147	3	3	5	
NH ₃ -N	0.6	2.8	0.4	0.3	0.2	0.6	3.1	---	---	---	
CHLORIDES	11	170	12	86	87	310	29	---	---	---	

--- All parameters in all samples underwent analysis. If concentration is not listed, the parameter was not detected above instrument detection limit.
J Estimated value.

Appendix C
CONTRACT LABORATORY PROGRAM
SPECIAL ANALYTICAL SERVICES

REQUEST FORMS

GLT821/15-2

U.S. Environmental Protection Agency
HWI Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[]

**SPECIAL ANALYTICAL SERVICES
Regional Request**

☒ Regional Transmittal

☐ Telephone Request

A. EPA Region and Site Name: Region V, NSL/ECC

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analyses for metals, chromium and copper per RAS protocol except with lower detection limits. Graphite furnace AA with Standard Addition method shall be used.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

Analyze 38 low level groundwater and leachate samples for the parameters listed above. All samples will be unfiltered.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund (Enforcement), Predesign.

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment: Daily by Overnight Carrier

METALS - page 2

- 6. Approximate number of days results required after lab receipt of samples:**

Laboratory should report results within 30 days of receipt of samples.

- 7. Analytical protocol required (attach copy if other than a protocol currently used in this program):**

Protocol as per Statement of Work for inorganics, 1987, SOW No. 787 except as noted in section 8. of this SAS.

- 8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

Detection limits for chromium - 2 ug/l and detection limits for copper - 2 ug/l.

- 9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.**

Same as the Statement of Work for inorganics, 1987, SOW No. 787.

- 10. Other (use additional sheets or attach supplementary information, as needed):**

- 11. Name of sampling/shipping contact:** Dave Shekoski
Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

METALS - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Chromium</u>	<u>2.0 ug/l</u>	<u>+/- 15%</u>
<u>Copper</u>	<u>2.0 ug/l</u>	<u>+/- 15%</u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>See Attachment I</u>	<u></u>	<u></u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

ATTACHMENT I

Special Instructions for GFAA and QC requirements for the analysis

1. Sample aliquots are preserved in the field as follows:

One liter sample preserved with 5 ml of 50% HNO₃ to pH <2 for for all metals except mercury.

2. Analysis of the two metals must be done by graphite furnace atomic absorption (GFAA) using method of standard additions for quantitation.
3. Samples for GFAA metals can be analyzed without digestion if the samples are clean and without any particulates. In this case, a calibration blank, duplicate, initial calibration verification standard (ICVS) and continuing calibration verification standard (CCVS) shall be analyzed without digestion.
4. If any of the samples contain particulate or significant suspended solids, sample aliquots, preparation blank, duplicate, matrix spikes and laboratory control samples are to be digested per ATTACHMENT II.
5. No identified field blank shall be used as a laboratory duplicate or matrix spike sample.

Appendix D
ISBH ANALYTICAL PROTOCOLS AND SAS FORMS

ISBH SAS FORMS

GLT821/15-9

U.S. Environmental Protection Agency
HWI Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[]

**SPECIAL ANALYTICAL SERVICES
Regional Request**

☒ Regional Transmittal

☐ Telephone Request

A. EPA Region and Site Name: Region V, NSL/ECC

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analyses for BOD₅ and COD. The analyses will be performed by the Indiana State Board of Health. This SAS request is being filled out to help document the analytical protocols used.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

Analyze 38 low level groundwater and leachate samples for the parameters listed above. All samples will be unfiltered.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund (Enforcement), Predesign.

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment: Daily by Overnight Carrier

BOD - page 2

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached ISBH method, ISBH Code No. BOD-A-2-88

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Two sample dilutions must overlap to result in a residual of D.O. 1 mg/l and a D.O. depletion of 2 mg/l. Results for 2 dilutions should agree within 15%. Prepare a seed correction bottle, a dilution water control in duplicate, and a glucose-glutamic acid check in addition to sample dilutions. Determine the initial and final D.O. of each bottle. Store samples at 4°C until analysis. The holding time is not to exceed 48 hours from time of sample collection. D.O. meter error is not to exceed 0.1 mg/l, 5 days apart. Use only the method specified above. The seed control sample should be run at 10 times the seed concentration. The result of the seed control samples should then be adjusted 1/10 before being used. Do not use blank results to calculate the seed concentration.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Submit all raw data. Report initial and final D.O. from each bottle. Report BOD in mg/l D.O. for each bottle and the average of each dilution fitting the depletion range listed above using calculations specified by "Standard Methods". Report results of duplicates, dilution water, control, seed control, and glucose-glutamic acid check. All records of analysis and calculations should be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>BOD</u>	<u>1.5 mg/l</u>	<u>10% or +/-5 mg/l</u>
_____	_____	_____

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Glucose-glutamic acid</u>	<u>1 per run of samples</u>	<u>160-240 mg/l</u>
<u>Duplicate</u>	<u>2 for runs < 10</u> <u>1 per 10 for runs <10</u>	<u>+/- 10%</u>
<u>Dilution water control</u>	<u>2 per batch of</u> <u>dilution water</u>	<u><0.2 mg/l</u>
<u>Seed control sample</u>	<u>2 per batch of</u> <u>dilution water</u>	<u>0.6 - 1.0 mg/l</u>
<u>EPA QC demand reference</u> <u>1 set of 2 ampules</u>	<u>1 per this project</u>	<u>80% - 120%</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

6. Approximate number of days results required after lab receipt of samples:

Laboratory shall report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

ISBH Method COD-B-10-88 (Storet No. 00335, Titrimetric method) will be used for COD less than 50 mg/L. ISBH method COD-A-10-88 will be used for COD greater than 50 mg/L. Samples will be preserved in the field with 2 ml of H_2SO_4 per liter of sample to pH less than 2.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Check sample pH (wide range pH paper). If pH > 2, contact LSSS, CRL for further instructions.
2. Use a) 50ml sample aliquots for both methods, b) 0.250 N $K_2Cr_2O_7$ reagent and 0.25 N ferrous ammonium sulfate titrant for Method 410.1, and c) 0.0250 N $K_2Cr_2O_7$ reagent and 0.025 N ferrous ammonium sulfate titrant for Method 410.2.
3. Dilute and reanalyze (by Method 410.1) any samples with COD values > 800 mg/l or titrant volumes < 5.0 ml. Reanalyze samples (by Method 410.1) if initial sample values are > 50 mg/l COD by Method 410.2. Reanalyze samples (by Method 410.2) if initial sample values are < 50 mg/l COD by Method 410.1.
4. Any sample aliquots < 50 mls will be diluted to 50 mls so that the COD reaction mixture will be 50% H_2SO_4 / 50% water by volume.
5. Titration blanks will be determined, at least in duplicate each day of analysis and will not differ more than +/-0.1 ml titrant for Method 410.1 and +/-1.0 ml titrant for Method 410.2.
6. Separate sets of QA Audits will be performed for each method, if both methods are used.
7. Use potassium hydrogen phthalate as a matrix spike compound. Use 20 mg/l matrix spike concentration for Method 410.2.
8. Samples will be refluxed for at least 2 hours.
9. Homogenize sample aliquots, as necessary, to obtain sample aliquots of representative suspended solids.
10. Use only the method specified.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records, tabulating titrant standardization, titration volumes for titration or sample blanks (2 or more in number), samples, and QA Audits will be provided for each method used. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA Audit results. Records of chloride analysis will be provided for any samples so specified on the RAS/SAS Traffic Report or SAS Packing List. Separate bench records will be provided for any COD determinations of high chloride samples (>2000 mg/l Cl) including weight of mercuric sulfate used, sample titration volume and titration blank volume for each sample type. EPA QC Reference samples, or any other reference samples, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

-
11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>COD low level</u>	<u>5 mg/l</u>	<u>+/- 5 mg/l</u>
<u>COD mid level</u>	<u>50 mg/l</u>	<u>+/- 10 mg/l</u>
<u> </u>	<u> </u>	<u> </u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
* Matrix spike 1	<u>2 for runs < 10</u> <u>1 per 10 for runs >10</u>	<u>80% - 120%</u>
<u>Duplicate</u>	<u>2 for runs < 10</u> <u>1 per 10 for runs >10</u>	<u>10% or 5 mg/l</u>
<u>Titration Blank (Used for calculation of results)</u>	<u>at lease 2 per sample set for each method used</u>	<u>difference in titrant volumes shall not exceed 0.1 ml</u>
<u>1 set of EPA QC Demand reference samples - at concentration levels</u>	<u>1 per sample set for each method used</u>	<u>90-110% recovery in aliquot tests</u>

* Matrix spike will be greater than 30% of the sample concentration, but spike sample shall not exceed the working range.

III. *Action Required if Limits are Exceeded:

Take corrective action and reanalyze samples. Contact Jay Thakkar (312)886-1972.

Contact Region V RSCC Jan Pels at (312)353-2720 concerning questions on chloride interferences and modifications of COD test.

Please return this request to the Sample Management office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

U.S. Environmental Protection Agency
HWI Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
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**SPECIAL ANALYTICAL SERVICES
Regional Request**

☒ Regional Transmittal

☐ Telephone Request

A. EPA Region and Site Name: Region V, NSL/ECC

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analyses for alkalinity, total suspended solids, total dissolved solids, volatile suspended solids, total Kjeldahl nitrogen, arsenic, nitrate/nitrite, ammonia, chlorides, total phosphorus, chromium (total and hexavalent), and sulfates. All samples will be run by the Indiana State Board of Health. This SAS request is being filled out to help document the analytical protocols used.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

Analyze 38 low level groundwater and leachate samples for the parameters listed above. All samples will be unfiltered.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund (Enforcement), Predesign

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment: Daily by Overnight Carrier

ALKALINITY - page 2

- 6. Approximate number of days results required after lab receipt of samples:**

Laboratory should report results within 30 days of receipt of samples.

- 7. Analytical protocol required (attach copy if other than a protocol currently used in this program):**

See attached method, ISBH Code No. Alk-B-1-88

- 8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

Samples should be stored at 4°C until analysis and validation of results. Sample holding time should not exceed 14 days from date of collection. Use potentiometric titration to pH 4.5 for alkalinity concentrations equal to or greater than 20 mg/l as CaCO₃. Do not use titrant volumes greater than 50 ml. Use only the method specified above.

- 9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.**

Test procedures used will be clearly identified. Bench records tabulating the order of titrant standardization, lab blanks, samples, lab control standard, spikes, duplicates, etc. with resulting titrant volume or titrant readouts will be provided along with copies of work sheets used to calculate results. All records of analysis and calculations must be legible.

- 10. Other (use additional sheets or attach supplementary information, as needed):**

- 11. Name of sampling/shipping contact:** Dave Shekoski

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ALKALINITY - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Alkalinity</u>	<u>3 mg/l for low level</u> <u>and 20 mg/l high level</u>	<u>+/- 10% for >20 mg/l</u> <u>CaCO₃ and +/-2 mg/l</u> <u>for <20 mg/l CaCO₃</u>
_____	_____	_____

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Sample spike</u>	<u>1 per run and 1 per 20 samples</u>	<u>85% - 115% recovery</u>
<u>Lab duplicate</u>	<u>1 per run and 1 per 10 samples</u>	<u>+/-10% for high level</u>
<u>Lab blank</u>	<u>1 per run and 1 per 10 samples</u>	<u><5 mg/l for high level</u> <u><2 mg/l for low level</u>
<u>EPA QC demand reference</u> <u>1 set of 2 ampules</u>	<u>1 per this project</u>	<u>80% - 115%</u>
<u>Titrant standardization</u>	<u>once each week</u>	

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone: (312) 353-2720)

Arsenic - page 2

- 6. Approximate number of days results required after lab receipt of samples:**

Laboratory shall report results within 30 days of receipt of samples.

- 7. Analytical protocol required (attach copy if other than a protocol currently used in this program):**

ISBH method attached, ISBH Code No. AS-A-3-87. Samples will be preserved in the field with 5 ml/L HNO₃ to pH < 2.

- 8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

Required detection limit is 0.2 ug/l. A five point calibration curve shall be used with one standard at 0 ppb and one standard at at the requested detection limit. One additional laboratory spike per matrix shall be run with the added concentration at the requested detection limit. The spike shall be added to the sample before sample preparation begins. Use only the method specified.

- 9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.**

Report results of all sample, spike, duplicate, blank and sample dilution analyses. Report all raw data including notebook entries, calculations, calibration results and sample dilutions.

- 10. Other (use additional sheets or attach supplementary information, as needed):**

-
- 11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426**

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

Arsenic - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Arsenic (As)</u>	<u>0.2 ug/l</u>	<u>+/- 15%</u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Method blanks</u>	<u>1 per 10 samples</u>	<u>sample detection limit</u>
<u>Spike analysis</u>	<u>1 per 20 samples and at least one per matrix</u>	<u>85% - 115% recovery</u>
<u>Duplicate analysis</u>	<u>1 per 20 samples and at least one per matrix</u>	<u>+/- 15%</u>
<u>Quality control laboratory standard</u>	<u>1 per project</u>	<u>+/- 15%</u>
<u>Laboratory spike (detection limit)</u>	<u>1 per matrix</u>	<u>sample detection limit</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

6. Approximate number of days results required after lab receipt of samples:

Laboratory will report results within 30 days after receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method, ISBH Code No. C1-C-1-88

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample will be kept at 4°C until analysis and validation of results. Dilute and rerun samples with absorbances higher than the highest standard. The holding time is not to exceed 28 days from the date of sample collection. Standards will be prepared daily from the stock solution. A minimum 5 point standard curve should be used. Use only method specified above.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify the method used. Bench records tabulating the order of titrant standardization, lab blanks, duplicates, samples, spikes, etc., with resulting titrant volumes or absorbance readings will be provided along with copies of worksheets used to calculate results. All records of analysis and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

CHLORIDES - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Chlorides</u>	<u>1.0 mg/l</u>	<u>+/- 10% for >10 mg/l</u> <u>or +/- 1 mg/l for</u> <u><10 mg/l and results</u> <u>to the nearest 1 mg/l</u> <u>and to 2 significant</u> <u>figures for > 10 mg/l</u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Lab duplicate</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>10% or 3 mg/l</u>
<u>Lab blank</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u><3 mg/l</u>
<u>EPA QC mineral ref.</u> <u>samples, 1 set of 2</u> <u>ampules</u>	<u>1 per this project</u>	<u>85% - 115%</u>
<u>Matrix spike 1</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>85% - 115%</u> <u>recovery</u>
<u>Calibration verifi-</u> <u>cation check sample</u>	<u>1 per 10 samples and</u> <u>beginning of each run</u>	<u>90% - 110%</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone: (312) 353-2720)

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed working range of the standard curve.

Chromium - hexavalent - page 2

6. Approximate number of days results required after lab receipt of samples:

Laboratory shall report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

ISBH method attached, ISBH Code No. H.Cr-A-1-88.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Required detection limit is 8 ug/l (0.008 mg/l). A five point calibration curve shall be used with one standard at 0 ppb and one standard at at the requested detection limit. One additonal laboratory spike per matrix shall be run with the added spike concentration at the requested detection limit. The spike shall be added to the sample before sample preparation begins. Samples will stored at 4°C and the allowable holding time is 24 hours.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Report results of all sample, spike, duplicate, blank and sample dilution analyses. Report all raw data including notebook entries, calculations, calibration results and sample dilutions.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

Chromium - hexavalent - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Hexavalent chromium</u> <u>(Cr)</u>	<u>0.008 mg/l (8 ug/l)</u>	<u>+/- 15%</u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Method blanks</u>	<u>1 per 10 samples</u>	<u>detection limit</u>
<u>Spike analysis</u>	<u>1 per 20 samples and at</u> <u>least one per matrix</u>	<u>85% - 115%</u> <u>recovery</u>
<u>Duplicate analysis</u>	<u>1 per 20 samples and at</u> <u>least one per matrix</u>	<u>+/- 15%</u>
<u>Quality control</u> <u>laboratory standard</u>	<u>1 per project</u>	<u>+/- 15%</u>
<u>Laboratory spike</u> <u>(detection limit)</u>	<u>1 per matrix</u>	<u>detection limit</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

Chromium - total - page 2

6. Approximate number of days results required after lab receipt of samples:

Laboratory shall report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

ISBH method attached, ISBH Code No. T.Cr-B-2-88. Samples will be preserved in the field with 5 ml/L HNO₃ to pH < 2.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Required detection limit is 8 ug/l (0.008 mg/l). A five point calibration curve shall be used with one standard at 0 ppb and one standard at at the requested detection limit. One additonal laboratory spike per matrix shall be run with the added concentration at the requested detection limit. The spike shall be added to the sample before sample preparation begins.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Report results of all sample, spike, duplicate, blank and sample dilution analyses. Report all raw data including notebook entries, calculations, calibration results and sample dilutions.

10. Other (use additional sheets or attach supplementary information, as needed):

-
11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

Chromium - total - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Total chromium (Cr)</u>	<u>0.008 mg/l (8 ug/l)</u>	<u>+/- 15%</u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Method blanks</u>	<u>1 per 10 samples</u>	<u>detection limit</u>
<u>Spike analysis</u>	<u>1 per 20 samples and at least one per matrix</u>	<u>85% - 115% recovery</u>
<u>Duplicate analysis</u>	<u>1 per 20 samples and at least one per matrix</u>	<u>+/- 15%</u>
<u>Quality control laboratory standard</u>	<u>1 per project</u>	<u>+/- 15%</u>
<u>Laboratory spike (detection limit)</u>	<u>1 per matrix</u>	<u>detection limit</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method, ISBH Code No. NH3-A-10-88

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample aliquots will be preserved with 2 ml/L H₂SO₄ to pH <2. Ammonia will be reported as mg/l N. Samples will be analyzed within 28 days after collection. Use a minimum 5 point standard curve (blank and 4 standards). Use only method specified.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedure used will be clearly identified. Bench records tabulating calibration standards, lab blanks, samples, lab control standards, etc. with resulting absorbance or concentration readouts will be provided along with copies of work sheets used to calculate ammonia results. All records of analyses and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

NITROGEN-AMMONIA - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Ammonia</u>	<u>0.1 mg/l</u>	<u>+/- 10 % for</u> <u>> 1 mg/l or</u> <u><0.1 mg/l for</u> <u>< 1 mg/l</u>

Report results to the nearest 0.05 mg/l and to 2 significant figures for concentrations exceeding 1 mg/l N.

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Lab duplicate</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>+/- 10% or 0.1 mg/l</u>
<u>Lab blank</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>0.1 mg/l</u>
<u>EPA QC nutrient ref.</u> <u>1 set of 2 samples</u>	<u>1 per this project</u>	<u>85% - 115%</u>
<u>Matrix spike¹</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>85% - 115% recovery</u>
<u>Laboratory control std.</u>	<u>1 per 10 samples and</u> <u>at the end of each run</u>	<u>85% - 115%</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V, (Phone (312) 353-2720).

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method, ISBH Code No. NO₃+NO₂ (N)-B-10-88

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample aliquots will be refrigerated until analysis and validation of results. Sample holding time will not exceed 28 days. Sample aliquots will be preserved with 2 ml/L H₂SO₄ to pH <2. Nitrate and nitrite will be reported as mg/l N. Use only method specified above. Use minimum 5 point standard curve.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedures used will be clearly identified. Bench records tabulating the order of calibration standards, lab blanks, samples, lab control standards, etc. with resulting absorbances of concentration readouts, will be provided along with copies of work sheets used to calculate results. Only one cadmium column should be used for an analytical run. If the column is changed, then the system must be recalibrated and a new set of audits is required. All records of analysis and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

-
11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Nitrate + Nitrite</u>	<u>0.10 mg/l as N</u>	<u>+/- 10% for > 1.0mg/l</u> <u>or +/- 0.1 mg/l for</u> <u>< 1.0 mg/l</u>

Results will be reported to the nearest 0.05 mg/l for conc. less than 1.0 mg/l and to 2 significant figures for concentrations exceeding 1.0 mg/l.

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Lab duplicate</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>10% or 0.1 mg/l</u>
<u>Lab blank</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>0.1 mg/l - N</u>
<u>EPA QC nutrient std.</u> <u>1 and 2 or 1 set of 2</u> <u>EPA, GC water supply</u> <u>nitrate samples.</u>	<u>1 per this project</u>	<u>85% - 115%</u>
<u>Matrix spike 1</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>85% - 115% recovery</u>
<u>Lab control check std.</u>	<u>1 per 10 samples</u> <u>beginning of each run</u>	<u>85% - 115%</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720.

1. Matrix spike concentrations will be greater than 30% of sample concentration but spiked sample will not exceed the working range of the standard curve.

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method, ISBH Code No. TKN-B-1-88

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample aliquots will be preserved in the field using 2 ml/L H₂SO₄ to pH <2 and should be kept at 4°C until analysis and validation of results. Report results as mg/l N. Holding time is not to exceed 28 days from the time of sample collection. Use only method specified above.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc. If not completed, format of results will be left to program discretion.

Copies of all bench records tabulating the duplicates, standards, lab blanks, lab control standard samples, sample results with absorbances and concentrations are to be reported and legible. Report results in mg/l N. Provide digestion logs showing sample aliquots and concentrations of all solutions tested.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
TKN	0.1 mg/l	+/- 10 % for > 1 mg/l or <0.1 mg/l for < 1 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab duplicate	1 per run and 1 per 10 samples	10% or 0.1 mg/l
Lab blank	1 per run and 1 per 10 samples	< 0.1 mg/l - N
EPA QC nutrient ref. samples, 1 set of 2 samples	1 per this project	85% - 115%
Matrix spike (org N)	1 per run and 1 per 10 samples	85% -115% recovery
Calibration standard	1 per 10 samples and end of set	85% - 115%

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V, (phone (312) 353-2720).

- Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.

6. Approximate number of days results required after lab receipt of samples:

Laboratory will report results within 30 days after receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method, ISBH Code No. P-A-1-88

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample aliquots will be preserved in the field using 2 ml/L H₂SO₄ to pH <2 and stored at 4°C until analysis and validation of results. Holding time is not to exceed 28 days from the time of sample collection. Use only method specified above. Use a minimum 5 point standard curve.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records and calculations for samples, blanks, duplicates, spikes, and all control checks with absorbances and concentrations will be provided with copies of the worksheets. Results to be reported as mg/l P.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Total phosphorous</u>	<u>0.03 mg/l</u>	<u>+/- 10 % for > 1.0 mg/l</u> <u>or +/- 0.1 mg/l</u> <u>for < 1.0 mg/l</u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Lab duplicate</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>10% or 0.1 mg/l</u>
<u>Lab blank</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>0.03 mg/l</u>
<u>EPA QC nutrient ref.</u> <u>samples, 1 set of</u> <u>2 samples</u>	<u>1 per this project</u>	<u>90% - 110%</u>
<u>Matrix spike</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>90% - 110%</u> <u>recovery</u>
<u>Calibration standard</u>	<u>1 per 10 samples and</u> <u>end of set</u>	<u>90% - 110%</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (phone (312) 353-2720).

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method, ISBH Code No. SF-A-1-88

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Use aliquots of 100 ml; however do not use sample aliquots yielding more than 200 mg of residue. Repeat analysis if residue is greater than 200 mg, using smaller aliquot. If pH is less than 4.0, raise pH value of aliquot to between pH 4 and 8 using NaOH. Subtract the weight of the sodium added from the weight of the residue. Samples will be kept at 4°C until analysis and validation of results. For TDS, the holding time is 7 days. Use only method specified above.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedure used will be clearly identified. Bench records tabulating weights used for calculations and to determine constant weight will be provided along with copies of work sheets used to calculate TDS results. All records and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Dissolved solids</u>	<u>20 mg/l</u>	<u>+/- 2 mg/l for</u>
		<u><200 mg/l</u>
		<u>or +/- 10%, >200 mg/l</u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Lab duplicate</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>+/- 2 mg/l, <200 mg/l</u> <u>+/- 10%, >200 mg/l</u>
<u>Lab blank</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>+/- 20 mg/l, 100 ml</u>
<u>EPA QC mineral ref.</u> <u>1 set of 2 samples</u>	<u>1 per this project</u>	<u>85% - 115%</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. SNF-A-1-88 using glass fiber filter discs without organic binder such as Millipore AP-40, Reeves Angel 934-AH, Gelman A/E, or equivalent. Membrane filter apparatus using 47 mm diameter glass fiber filter and coarse (40-60) micron fritted disc as filter support must be used. The filter and support specifications are mandatory. Sample will be collected in a one liter bottle and must be kept at 4°C until data are validated. Holding time is 7 days from date of collection.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Do not filter more than a 200 ml sample aliquot.
2. Duplicate sample aliquots will be filtered with two or more intervening samples.
3. Aliquot filtered should provide residue greater than 1.0 mg for aliquots less than 200 ml.
4. Residues are to be weighed to constant weight pursuant to "Standard Methods" Part 7.1, Method 160. Final weight is to be used for calculations.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records of tare weights, final weights, volumes filtered, order of blanks, duplicates, samples filtered will be provided along with copies of worksheets used to calculate results. Specify manufacturer type and diameter (mm) of glass fiber filter used. All records of analysis and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Suspended solids</u>	<u>2 - 3 mg/l for 200 ml</u>	<u><0.5 mg for</u> <u><50 mg/l or</u> <u>+/- 10% for</u> <u>>50 mg/l</u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Lab duplicate</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>+/- 0.5 mg, < 50 mg/l</u> <u>+/- 10%, > 50 mg/l</u>
<u>Lab blank</u>	<u>1 per run and 1 per</u> <u>10 samples</u>	<u>+/- 0.5 mg</u>
<u>EPA QC residue ref.</u> <u>1 set of 2 samples</u>	<u>1 per this project</u>	<u>+/- 5 mg/l, < 50mg/l</u> <u>+/- 10%, >50 mg/l</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720).

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached ISBH method, ISBH Code No. SV-A-4-87. Holding time is 7 days from date of collection. Store at 4°C.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Furnace must be up to temperature before inserting sample.
2. Do not overload desiccator.
3. Continue to dry and weigh the sample until there is less than 0.5 mg difference between successive weighings.
4. Use TSS filtered residues for analysis.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records of tare weights, final weights, order of blanks, duplicates will be provided along with copies of worksheets used to calculate results. Specify manufacturer type of muffle furnace. All records of analysis and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

-
11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

VSS - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Suspended solids</u>	<u>2-3 mg/l for 200 ml</u>	<u><0.5 mg for duplicates</u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
<u>Lab duplicate</u>	<u>1 per run and 1 per 10 samples</u>	<u>+/- 0.5 mg</u>
<u>Lab blank</u>	<u>1 per run and 1 per 10 samples</u>	<u>+/- 0.5 mg</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720).

6. Approximate number of days results required after lab receipt of samples:

Laboratory will report results within 30 days after receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method, ISBH Code No. SO₄-B-1-88

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Dilute and rerun samples with absorbances higher than the highest standard. The holding time is not to exceed 28 days from the date of sample collection. Samples will be stored at 4°C. Standards will be prepared daily from the stock solution. Use only the method specified.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedures will be clearly identified. Bench records tabulating the calibration standards, lab blanks, duplicates, samples and spikes will be provided along with copies of worksheets used to calculate results. All records of analysis and calculations must be legible. Report results in mg/l SO₄.

10. Other (use additional sheets or attach supplementary information, as needed):

-
11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

SULFATES - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
<u>Sulfates</u>	<u>3 mg/l methylthymol blue</u>	<u>+/- 2 mg/l for <20 mg/l</u>

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits (+/- % or conc.)
<u>Lab duplicate</u>	<u>1 per run and 1 per 10 samples</u>	<u>+/- 10% or 2 mg/l</u>
<u>Lab blank</u>	<u>1 per run and 1 per 10 samples</u>	<u>< 3 mg/l</u>
<u>EPA QC mineral ref. 1 set of 2 samples</u>	<u>1 per this project</u>	<u>85% - 115%</u>
<u>Matrix spike ¹</u>	<u>1 per run and 1 per 10 samples</u>	<u>85% - 115% recovery</u>
<u>Continuing calibration check</u>	<u>1 per 10 samples and beginning of each run</u>	<u>90% - 110%</u>

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (phone (312) 353-2720).

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.

ISBH QA/QC

GLT821/15-7

CONTROL OF ANALYTICAL PERFORMANCE

1. General

It is assumed that a valid sample has been properly taken, preserved, and delivered to the laboratory. It is also assumed that the analytical methods used are carefully documented and meet the following criteria:

- a. The methods should measure the desired constituent with precision and accuracy sufficient to meet the data needs in the presence of the interferences normally encountered in polluted waters.
- b. The procedure should utilize the equipment and skills normally available in the average water pollution control laboratory.
- c. The selected methods should be in use in many laboratories or have been sufficiently tested to establish their validity.
- d. The methods should be sufficiently rapid to permit routine use for the examination of large numbers of samples.

In order to evaluate daily performance to document that valid data are being produced, it is necessary that valid precision and accuracy data be available on the method and analyst. Thereafter, systematic daily checks are required to show that reproducible results are being obtained and that the methodology is actually measuring what is in the sample.

2. Daily Performance Audits

Once valid precision and accuracy data are available for the method and the analyst, systematic daily checks are necessary to insure that valid data are being obtained. The following aspects of evaluation are used in the laboratory:

- a. Blanks: Distilled-deionized water, with the appropriate preservative, is analyzed with each daily sample run to determine and monitor laboratory detection limits for each parameter. The frequency of blank runs is at least one per set of samples or one in 20 samples.
- b. Control Standards: A stock solution of known concentration is prepared and made available to the chemists for daily analysis. A control standard solution should be run with every 10-20 field samples and the results recorded on a Quality Control Data Sheet (Attachment 13).

The control standards are summarized bimonthly to prepare control limits for each parameter. Each chemist receives the control limits (Attachment 14) which gives the mean, true value, standard deviation, warning limits, and the rejection limits for each parameter.

Since the quality control samples are run with a frequency of approximately 5 percent, the rejection limits give guidelines to the chemist as to the performance of the method and necessary action to be taken by the analyst when he obtains out of control results.

If the result of the quality control solution is between ± 2 and ± 3 standard deviations from the mean value, the chemist is directed to check the procedure for such things as calibration, quality of standards, quality of reagents, and analytical techniques. A difference from the mean value of the control sample equal or greater than ± 3 standard deviations indicates the analytical run is out of control and should be stopped. Since the sample results are considered unreliable, a report to the laboratory quality control officer is made and a complete evaluation of the analytical procedure should be made. After satisfactory quality control samples are obtained, the samples should be rerun.

- c. Duplicate analysis: In order to show that reproducible results are being obtained, it is necessary to run duplicate samples on each run. It is a good practice to run duplicate samples, selected to cover different types of samples and various concentration ranges, at least 5 percent of the time. All results are recorded on the Quality Control Data Sheet (Attachment 13) as previously mentioned with the control samples. The duplicate analyses are reported monthly and summarized every six months to prepare results showing the concentration range, sample type, mean value of the determinations, standard deviation, and the 95 percent confidence range. This summary will provide the basis to judge precision of the analytical run for samples of a certain type and range.

The actual daily control limits for precision is prepared every six months and given to the analyst (Attachment 15-A). The data provides a means of placing a control limit on each set of duplicate analyses. Using results from past duplicate analyses, the precision of routine sample analyses is indicated by the calculation of the Shewhart upper control limit (UCL) for a concentration range R . The upper control limit is used as a critical range value (R_c) or the largest difference between duplicate analyses in a certain range which is acceptable.

$$R_c = UCL = D_4 R$$

D_4 = Shewhart factor for ranges based on duplicate analyses

$$= 3.27$$

R = average for a designated range

The difference in duplicate sample results is compared to the R_c for a certain parameter range. If the difference is greater than the critical range value (R_c), then the precision is out of control and the analytical problem is reported to the laboratory quality assurance officer and the analysis is rerun.

(Reference: 1979 EPA Handbook for Analytical Quality Control In Water and Wastewater Laboratories)

The results of blanks, daily duplicates, and the control standards are recorded in such a manner that they are available for review. All results of control standards, duplicate results, spikes and blanks are stored in a computer. This enables the laboratory to efficiently handle the required statistical analysis for precision and accuracy calculations.

- d. Spiked Sample Analysis: Samples which are received in the laboratory are split, and one aliquot is spiked and analyzed for the parameter of interest at a frequency of one in 20 samples. The amount of spiking material which is used is five times the detection limit for low values or twice the known concentration of the parameter of concern for concentrations higher in the working range. The recovery of the spiked material indicates the matrix affect on the analytical data. The laboratory expects a recovery of the inorganic parameters from 80 percent to 120 percent and recovery of organic spiked parameters in the suggested ranges designated by EPA.

3. Field Quality Control

The field blank is a sample which is submitted to the laboratory to give an indication of the integrity of the sample containers, preservative, field glassware, and sample collection techniques. The analytical results will provide information on possible contamination prior to laboratory receipt of the samples.

The field duplicates are two separate samples taken at the same field location. They serve the purpose of showing accuracy and precision in the field sampling techniques.

DATE PRINTED 11-14-87

-9/-

FORM A17
Environmental Labs
LABORATORY QC REPORTING SHEET
METAL ANALYSIS
TASK 1

LAB ID# _____
FIELD ID# _____

ELEMENT	DATE	LAB SLANK		DUPLICATE				QC SOLUTIONS				NOTES		
		#1	#2	SAMPLE #	#1a	#1b	SAMPLE #	#2a	#2b	TRUE	#2		#b	#c
ARSENIC														
(ug/l)	/ /									12				
	/ /									12				
	/ /									12				
BARIUM														
(ug/l)	/ /									400				
	/ /									400				
	/ /									400				
CADMIUM														
(ug/l)	/ /									200				
	/ /									200				
	/ /									200				
CHROMIUM														
(ug/l)	/ /									200				
	/ /									200				
	/ /									200				
LEAD														
(ug/l)	/ /									300				
	/ /									300				
	/ /									300				
MERCURY														
(ug/l)	/ /									2.00				
	/ /									2.00				
	/ /									2.00				
NICKEL														
(ug/l)	/ /									300				
	/ /									300				
	/ /									300				
SELENIUM														
(ug/l)	/ /									12.0				
	/ /									12.0				
	/ /									12.0				
SILVER														
(ug/l)	/ /									200				
	/ /									200				
	/ /									200				

NOTES

- 1 - SPIKE RECOVERY DATA ATTACHED.
 2 - STANDARD ADDITION DATA ATTACHED.
 3 - DIGESTED QC RESULTS ATTACHED.
 4 - ADDITIONAL DATA LISTED BELOW.
 5 - SAMPLES DIGESTED BY METHOD 3010.
 6 - SAMPLES DIGESTED BY METHOD 3050.
 7 - SAMPLES TREATED BY U.S.E.P.H. CONCENTRATION METHOD
 (EPA, TOTAL RECOVERABLE METALS-4.1.4)
- 1 - AUTOMATED HYDRIDE METHOD OR COLD VAPOR.
 2 - DIRECT ASPIRATION OF SAMPLES.
 3 - QC AND STANDARDS CARRIED THROUGH
 ENTIRE SAMPLE TREATMENT PROCESS.
 4 - STANDARDS NOT CARRIED THROUGH SAMPLE TREATMENT.
 5 - QC WAS USED AS A CHECK STANDARD.

- 93 -

LABORATORY QC REPORTING SHEET
MUT. ANALYSIS
CASE 1

FIELD ID#

[illegible]

HAST
DEF. 18 AUGUST 1960
177 PAGE

HINDU

BLANK WAS RUN WITH
SAMPLE GROUP

RESULT (PFB)

PRETREATMENT

DATE _____

- 96 -

DATE	TIME	LOCATION	WIND	TEMP	SEA	REMARKS
1964	0000	10-10-64	000	10.0	000	10-10-64
1964	0100	10-10-64	000	10.0	000	10-10-64
1964	0200	10-10-64	000	10.0	000	10-10-64
1964	0300	10-10-64	000	10.0	000	10-10-64
1964	0400	10-10-64	000	10.0	000	10-10-64
1964	0500	10-10-64	000	10.0	000	10-10-64
1964	0600	10-10-64	000	10.0	000	10-10-64
1964	0700	10-10-64	000	10.0	000	10-10-64
1964	0800	10-10-64	000	10.0	000	10-10-64
1964	0900	10-10-64	000	10.0	000	10-10-64
1964	1000	10-10-64	000	10.0	000	10-10-64
1964	1100	10-10-64	000	10.0	000	10-10-64
1964	1200	10-10-64	000	10.0	000	10-10-64
1964	1300	10-10-64	000	10.0	000	10-10-64
1964	1400	10-10-64	000	10.0	000	10-10-64
1964	1500	10-10-64	000	10.0	000	10-10-64
1964	1600	10-10-64	000	10.0	000	10-10-64
1964	1700	10-10-64	000	10.0	000	10-10-64
1964	1800	10-10-64	000	10.0	000	10-10-64
1964	1900	10-10-64	000	10.0	000	10-10-64
1964	2000	10-10-64	000	10.0	000	10-10-64
1964	2100	10-10-64	000	10.0	000	10-10-64
1964	2200	10-10-64	000	10.0	000	10-10-64
1964	2300	10-10-64	000	10.0	000	10-10-64
1964	2400	10-10-64	000	10.0	000	10-10-64
1964	2500	10-10-64	000	10.0	000	10-10-64
1964	2600	10-10-64	000	10.0	000	10-10-64
1964	2700	10-10-64	000	10.0	000	10-10-64
1964	2800	10-10-64	000	10.0	000	10-10-64
1964	2900	10-10-64	000	10.0	000	10-10-64
1964	3000	10-10-64	000	10.0	000	10-10-64
1964	3100	10-10-64	000	10.0	000	10-10-64
1964	3200	10-10-64	000	10.0	000	10-10-64
1964	3300	10-10-64	000	10.0	000	10-10-64
1964	3400	10-10-64	000	10.0	000	10-10-64
1964	3500	10-10-64	000	10.0	000	10-10-64
1964	3600	10-10-64	000	10.0	000	10-10-64
1964	3700	10-10-64	000	10.0	000	10-10-64
1964	3800	10-10-64	000	10.0	000	10-10-64
1964	3900	10-10-64	000	10.0	000	10-10-64
1964	4000	10-10-64	000	10.0	000	10-10-64
1964	4100	10-10-64	000	10.0	000	10-10-64
1964	4200	10-10-64	000	10.0	000	10-10-64
1964	4300	10-10-64	000	10.0	000	10-10-64
1964	4400	10-10-64	000	10.0	000	10-10-64
1964	4500	10-10-64	000	10.0	000	10-10-64
1964	4600	10-10-64	000	10.0	000	10-10-64
1964	4700	10-10-64	000	10.0	000	10-10-64
1964	4800	10-10-64				

DATE PRINTED :16-Apr-87

-98-

FORM 454
Environmental Labs
LSBH

QUALITY CONTROL DATA SHEET

PARAMETER

MONTH OF

QC SOLUTIONS USED

T.V

UNITS

DATE	QC NO.	RESULT	ANALYST	DATE	QC NO.	RESULT	ANALYST
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P E R C I S I O N D A T A

DATE	SAMPLE NO	SAMPLE TYPE	RESULT A	RESULT B	Diff.	% OF RUN	ANALYST
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201 ALL CHEMISTS
WATER AND SEWAGE LABORATORY DIVISIONS

SUBJECT: QUALITY CONTROL

STANDARD DEVIATION(S) HAS BEEN CALCULATED FROM THE ANALYSIS OF A FIXED VALUE CONTROL SAMPLE.

WARNING LIMITS (WL):

A DIFFERENCE FROM THE MEAN VALUE OF THE CONTROL SAMPLE EQUAL TO $\pm 3s$, WHEN THE VALUE OF THE CONTROL SAMPLE IS LESS THAN OR MORE THAN THE MEAN VALUE $\pm 1s$. THE PROCEDURE SHOULD BE CHECKED FOR SUCH THINGS AS CALIBRATION, QUALITY OF THE STANDARDS, QUALITY OF THE REAGENTS AND ANALYTICAL TECHNIQUES.

CONTROL LIMITS (CL):

A DIFFERENCE FROM THE MEAN VALUE OF THE CONTROL SAMPLE EQUALS $\pm 1s$. WHEN THE VALUE OF THE CONTROL SAMPLE IS LESS THAN OR MORE THAN THE MEAN VALUE $\pm 1s$ THE ANALYSIS IS OUT OF CONTROL. THE RUN SHOULD BE STOPPED AND THE SAMPLE RESULTS CONSIDERED UNRELIABLE. A COMPLETE EVALUATION OF THE ANALYTICAL PROCEDURE SHOULD BE MADE, AND AFTER SATISFACTORY QUALITY CONTROL SAMPLES ARE OBTAINED, THE SAMPLES SHOULD BE RE-RUN.

CONTROL LIMITS ON QUALITY CONTROL SAMPLES

DETERMINATION	DESIGNAT	VALUE	RUNS	AVG.	STD. DEVIATION	FROM	TO	FROM	TO	NOTES
CHROMIUM	M251CP	900	29	374	27	820	928	755	955	
CHROMIUM-A	M2	115	15	5.4	0.1	5.2	5.6	5.1	5.7	
CHROMIUM-B	M01	100.0	5	10.1	0.2			9.1	11.1	*
ANTIMONY	M29	15	5	14.2	0.6	13.0	15.3	12.5	15.8	**
ARSENIC	M29	15.0	49	11.9	0.4	11.1	12.7	10.7	13.1	
ARSENIC	M11CP	12						11.9	14.5	**
BARIUM	M5	400	19	441	12	417	465	405	477	
BARIUM	M61CP	400	16	446	14	418	474	404	468	
BERYLLIUM	M251CP	200	14	199	15	157	231	151	247	
BERYLLIUM	M251CP	200	47	205	9	190	226	181	235	
BORON	M251CP	1000						982	1026	**
BORON	M241CP	200	4	190	9	172	208	163	217	
BORON	M24	200	41	204	3	192	210	195	213	
BROMINE	M2	50	4	37	1	31	35	30	36	
CADMIUM	M251CP	26						25.1	26.7	**
CADMIUM	M2	20.0	20	19.6	0.5	18.6	20.6	18.1	21.1	
CHLORINE	M81	100	17	96	7	90	102	85	105	
CHLORINE-TOTAL	M221CP	200	3	206	11			185	227	*
CHLORINE-TOTAL-M4	M20	200	25	207	9			186	228	*
CHLORINE-TOTAL-COLONGE2	M20	200	9	200	5	193	215	188	218	
COPPER	M251CP	200	4	230	5	228	231	223	237	**
COPPER-TOTAL	M251CP	200	3	215	3	202	228	202	232	
COPPER-TOTAL	M2	20	11	21	1	20	22	19	24	
COPPER-TOTAL	M2	20	13	20	1	19	21	18	22	
COPPER-TOTAL	M2	20	12	0.025	0.001	0.024	0.025	0.020	0.026	
COPPER-TOTAL	M2	20	15	2.5	0.1	2.2	2.7	2.0	3.0	

Attachment -14

10-20-67 DATE

CONTROL LIMITS ON QUALITY CONTROL SAMPLES

DETERMINATION	QC DESIGNATION	TRUE VALUE	NO. RUNS	AVG. STANDARD VALUE DEVIATION	WARNING LIMITS TO FROM	CONTROL LIMITS TO FROM	NOTES
FLUORIDE	M1	1.0	117	1.1	0.3	1.0	+
CHLORIDES	M2	50	17	51	1	73	+
IRON	M24	500	57	502	248	321	885
LEAD	M24	500	42	299	13	256	559
AMMONIUM	M2210P	1.5				1.67	1.19
PHOSPHORUS	M2110P	200				179	221
PHOSPHORUS	M2110P	200				183	219
AMMONIUM	M24	200	43	201	5	210	189
MERCURY	M23	2.0	18	2.3	0.1	2.6	2.7
AMMONIUM	M25	300				357	391
AMMONIUM	M2410P	300				321	371
AMMONIUM	M24	300	35	314	11	336	357
NITRATE-NITRITE-N	M2	2.9	23	2.8	0.1	3.1	3.1
OIL AND GREASE	M2	20.1	9	20.8	0.3	13.7	22.9
PHENOL	M2A	10.0	5	9.3	0.5	8.3	10.3
PHENOL	M2	40.0	5	38.2	0.8	34.4	42.0
PHOSPHORUS	M2	0.50	34	0.50	0.01	0.45	0.55
PHOSPHORUS	M13	4.5	24	5.4	0.2	4.8	6.0
PHOSPHORUS	M25	15.0	32	14.0	0.9	12.1	15.1
PHOSPHORUS	M2710P	400	2	371	2	382	400
SILICON	M2710P	1000	2	977	35	922	1011
SILICON	M27	200	37	207	3	186	228
SILICON	M18	25.2	10	27.7	0.9	21.9	33.4
SPECIFIC CONDUCTANCE	M2	700	3	557	32	591	723
STRONTIUM	M30	900	6	924	12	832	1016
STRONTIUM	M2210P	900	6	907	3	816	998
SULFATE	M6	50	20	60	1	54	66
SULFATE	M25	750	9	772	33	704	832
TALLIUM	M17	990	7	1080	23	972	1188
TIN	M4	4.0	22	4.0	0.2	3.7	4.3
TIN	M4	13.1	15	13.0	0.5	11.5	14.5
TOTAL DISSOLVED SOLI	M9	200	7	204	+	184	224
TOTAL SOLIDS	M9	200	25	202	+	182	222
AMMONIUM	M25	300	4	295	2	266	324
ZINC	M2410P	200	55	205	5	198	212
ZINC	M24	200	30	203	+	183	223

* 1% OR 35 AVG. VALUE
** PREVIOUS QC DATA

DATE: January 16, 1984

TO: All Chemists

SUBJECT: QUALITY CONTROL - PRECISION DATA (Liquid Samples)

Data obtained from duplicate analyses of samples which were submitted from January 1979 to June 1983, were used to calculate control limits which were expressed as R_c (critical range values). If the difference in the duplicate sample results is greater than the R_c value, then the run should be stopped and the sample results considered unreliable. The analytical procedure should be evaluated and the samples should be rerun.

CONTROL LIMITS ON DUPLICATE SAMPLE ANALYSIS

<u>Parameter</u>	<u>Avg.</u>	<u>Number of Duplicates</u>	<u>R_c</u>
Alkalinity			
1. - 200.	132.	167	3.5
200. - 400.	283.	291	5.8
Aluminum			
20. - 100.	40.	10	8.
100. - 1000.	390.	19	65.
Arsenic			
1. - 10.	2.9	184	1.0
10. - 100.	28.1	28	6.4
BOD			
1. - 10.	3.7	513	1.2
10. - 50.	23.8	272	5.9
50. - 150.	84.4	115	16.8
150. - 300.	205.	41	45.8
300. - 1000.	519.	35	109.
Cadmium			
10. - 50.	29.5	20	5.5
50. - 100.	64.2	5	6.9
100. - 500.	301.	16	72.6
500. - 1000.	791.	9	521.
Calcium			
1. - 50.	26.7	4	--
50. - 200.	142.	150	5.0
200. - 400.	243.	129	5.6
Chloride			
1. - 10.	7.0	77	1.4
10. - 100.	36.3	687	1.6
100. - 500.	180.	87	9.2
Chrome, Hex			
10. - 100.	11.	2	--
100. - 1000.	230.	2	82.

at low limit 15-A

Chrome, Total-Color			
10. - 100.	40.	57	13.
100. - 1000.	260.	57	47.
Chrom, Total, AA			
10. - 100.	22.4	39	18.4
100. - 500.	245.	20	53.5
500. - 1000.	0.0	0	0.0
1000. - 5000.	2306.	4	613.
COD			
5. - 50.	21.6	444	5.8
50. - 150.	91.2	88	10.3
150. - 300.	222.	83	24.2
Copper			
10. - 100.	36.0	147	8.4
100. - 500.	218.	52	16.
Cyanide			
.0050 - 0.1000	0.2	137	0.010
.1000 - 0.5000	.23	21	.058
.5000 - 1.0	.68	4	.12
.1 - 10.	2.2	4	.59
Fluoride			
0.10 - 0.50	.27	319	0.02
.50 - 1.	.80	738	0.04
1. - 5.	2.2	1199	0.13
Hardness			
1.0 - 100.	48.8	35	4.2
100. - 200.	152.	80	4.9
200. - 400.	305.	416	6.8
400. - 1000.	494.	72	8.9
Iron			
100. - 1000.	507.	207	75.9
1000. - 2000.	1455.	137	133.
2000. - 5000.	3030.	112	392.
Lead			
20. - 100.	40.9	54	12.1
100. - 400.	207.	28	21.4
400. - 1000.	634.	10	374.
Manganese			
20. - 100.	61.4	167	11.
100. - 500.	209.	232	19.
500. - 1000.	680.	33	58.
Mercury			
0.10 - 0.50	0.18	83	0.13
.50 - 1.00	.63	13	0.28
1. - 3.	1.8	16	0.66

Nickel			
20. - 100.	44.5	72	18.0
100. - 500.	224.	47	21.3
500. - 1000.	667.	16	69.3
Nitrogen-Ammonia			
0.10 - 0.50	0.21	267	0.10
0.50 - 1.00	0.66	80	0.10
1.00 - 5.00	2.5	191	.13
5.00 - 10.00	7.5	79	.25
Nitrogen-Nitrate + Nitrate			
0.1 - 0.5	0.27	175	0.07
.5 - 1.0	0.70	128	0.09
1.0 - 5.0	2.4	479	0.24
5.0 - 10.0	6.9	131	0.78
TKN			
0.1 - 1.0	0.59	296	0.21
1.0 - 5.0	2.1	193	0.45
5.0 - 10.0	7.0	26	0.70
10.0 - 50.	20.1	33	1.6
Phenol			
0.0050 - 0.0500	.01	12	0.006
.0500 - 0.10	.07	4	0.014
.10 - 1.00	.29	3	0.41
1.00 - 10.00	3.7	27	2.0
Phosphorous			
0.03 - 0.10	0.06	157	.03
.10 - 0.50	0.21	285	.05
.50 - 1.00	0.70	60	.13
1.00 - 5.0	2.6	137	.34
Potassium			
1. - 5.	2.3	318	0.27
5. - 10.	6.4	43	.47
10. - 50.	15.2	9	3.0
Silver			
1.00 - 1000.	10.0	35	8.3
Sodium.			
1. - 10.	6.4	148	0.9
10. - 50.	21.3	239	3.1
50. - 100.	70.5	33	7.6
Suspended Solids			
1. - 10.	5.6	177	2.7
10. - 50.	24.1	391	7.9
50. - 150.	84.0	181	21.1
150. - 500.	255.	78	72.8

Total Solids			
1.00 - 100.	57.1	36	3.6
100. - 500.	343.	174	26.7
500. - 1000.	682.	186	41.0
1000. - 5000.	1895.	108	207.
Sulfate			
1. - 50.	31.2	257	4.4
50. - 100.	68.5	327	5.8
100. - 200.	132.	71	18.6
200. - 500.	347.	32	35.5
TOC			
1. - 5.	3.3	93	1.1
5. - 10.	7.3	121	2.0
10. - 25.	15.2	98	4.4
25. - 200.	75.0	100	29.4
Zinc			
20. - 100.	42.8	150	6.9
100. - 500.	222.	85	11.5
500. - 1000.	746.	14	43.9
1000. - 5000.	2896.	28	233.

-104-

PRECISION & ACCURACY DATA
SPIKE STUDIES

ISBH Code No. _____
Storet No. _____

Determination: _____

Sample Source: _____

Chemist: _____

Date: _____

Runs	Sample	
	As Rec'd.	_____ mg/l Added
1		
2		
3		
4		
5		
6		
7		

Mean: _____
s: _____
C.V.: _____
% Recovery: _____
Relative Error: _____

Remarks:

[illegible]

MERCURY

[illegible]

INTERLABORATORY AUDITS

Analyses on spiked samples are made quarterly to give recovery data that is utilized in the validation of analytical performance. Concentrated solutions of a known value are obtained from EPA and are used to prepare spiked samples. The spiked samples include distilled water and natural sample matrices which are spiked with the concentrated solutions. The results of the recovery data are documented and maintained by the laboratory quality assurance officer.

The laboratory participates in the EPA, International Joint Commission, and other performance evaluation programs which offer forms of audit for the agency's quality assurance program. All results are maintained in the laboratory and utilized in the validation of the laboratory results.

ISBH ANALYTICAL PROTOCOLS

GLT821/15-8

ALKALINITY, TOTAL
(EPA Method, 1983)

ISBH Code No. Alk-B-1-88
STORET No. 00410
Approved for NPDES

1. Scope and Application

- 1.1 This method is applicable to drinking waters and surface waters, domestic and industrial wastes, and saline waters.
- 1.2 The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 ml.

2. Summary of Method

- 2.1 An unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way.

3. Sample Handling and Preservation

- 3.1 The sample should be refrigerated to 4° C and run as soon as possible.
- 3.2 Do not open the sample before analysis. The maximum holding time (per Methods Manual, EPA Vol. 49, No. 209, October 1984) is 14 days.

4. Comments

- 4.1 Substances such as weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.
- 4.2 For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in Annual Book of ASTM Standards, Part 31, Water, p. 129, D 1067, Method D, 1976.
- 4.3 Oil and grease, by coating the pH electrode, may interfere, causing sluggish response.

5. Apparatus

- 5.1 pH meter.
- 5.2 pH electrodes.
- 5.3 Magnetic stirrer, pipets, flasks, and other standard laboratory equipment.
- 5.4 Buret, Pyrex, 25 ml.

6. Reagents

6.1 Standard sulfuric acid, 0.02 N.

6.2 Standard sulfuric acid, 0.1 N.

7. Procedure

7.1 Sample size and titrant

7.1.1 Use 50 ml sample or some convenient aliquot to obtain 50 ml of titrant or less.

7.1.2 For alkalinity of 1000 mg CaCO_3 /l, use 0.02 N titrant (6.1)

7.1.3 For alkalinity of 1000 mg CaCO_3 /l, use 0.10 N titrant (6.2)

7.2 Potentiometric titration

7.2.1 Place sample in a 150 ml beaker by pipetting with pipet tip near the bottom of the beaker.

7.2.2 Measure pH of sample.

7.2.3 Add standard acid (6.1 or 6.2), being careful to stir thoroughly but gently to allow needle to obtain equilibrium.

7.2.4 Titrate to pH 4.5 and record volume.

8. Calculations

8.1 Alkalinity, as mg/l CaCO_3 /l = $\frac{A \times N \times 50,000}{\text{ml of sample}}$

Where: A = ml standard acid.

B = normality of standard acid.

9. Quality Control

9.1 One of every 20 samples is run in duplicate for use as precision data.

9.2 One of every 20 samples is spiked to evaluate the method for accuracy audits.

9.3 Internal audits using USEPA ampules.

9.4 Interlaboratory studies sponsored by USEPA.

10. References

- 10.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 269, Method 403, 1975.
- 10.2 Annual Book of ASTM Standards, Part 31, Water, p. 129, D 1067, Method E, 1976.
- 10.3 Methods for Chemical Analysis of Water and Wastes, EPA, p. 310-1, 1983.

ENHABSN
2-2-88
NAC
ALKALIC TXT

ARSENIC
(Manual Digestion, Automated Hydride Generation)
ISBH Environmental Laboratory Division Procedure, 1987

ISBH Code No. As-A-3-87
Storet No. Total 01002
Dissolved 01000
Suspended 01001
Approved for NPDES and SDWA

1. Scope and Application

- 1.1 The gaseous hydride method is applicable to most surface and ground water and samples with a moderate amount of suspended solids. Samples containing considerable suspended or organic material and soil samples may be analyzed directly by diluting them or they may be pre-digested or leached prior to analysis.

2. Summary of Method

- 2.1 Samples are treated with persulfate and acid and autoclaved at 121 degrees C to oxidize any organic arsenic compounds present. Hydrochloric acid is added to each sample, and the samples are then analyzed using an automated system. Sodium borohydride reagent converts arsenic present to arsine (AsH_3), which is separated from the solution by the evolved hydrogen gas and aeration with nitrogen. The generated arsine is swept into an electrically heated silica tube in the light path of an atomic absorption spectrophotometer, where it is broken down to arsenic atoms and detected at the 193.7 nm wavelength. The AA output is sent both to a chart recorder and to an A/D converter in the HP Laboratory Automation System sample loop. The working range of the method is 0.2-24 ug/l.

3. Sample Handling and Preservation

- 3.1 Water samples are preserved at the time of collection by the addition of 5 ml. of conc. nitric acid per liter. Samples are usually collected in plastic bottles but they may be collected in glass jars. Thicker samples (sludges, soil, or sediments) and highly caustic samples require no preservative.

4. Interferences

- 4.1 Organic forms of arsenic are converted to inorganic As by acid-persulfate treatment and autoclaving. Other digestion methods such as heating with $\text{HNO}_3/\text{H}_2\text{SO}_4$ are not effective in breaking down methylated As compounds (ref. 10.11). The persulfate-autoclave digestion method will tolerate at least 80 mg/l TOC levels and give complete digestion of cacodylic acid (dimethylarsinic acid) using approximately 0.2 g of potassium persulfate for a 30 ml. sample. At higher TOC levels, cacodylic acid is not completely digested, but some other less stable organo-arsenic compounds are still effectively decomposed.

- 4.2 At the hydrochloric acid and sodium borohydride concentrations used in the automated hydride generation procedure, As(III) is 20-30 percent more sensitive than As(V). However, after the persulfate-autoclave digestion, all As is converted to As(V) in both standard and sample solutions and so the arsine generation occurs from a single ionic species.
- 4.3 Some matrix components at high concentrations may cause partial or even total suppression of arsine. Possible interferences include noble and transition metals (notably Cu and Ni), hydride forming elements such as Se, etc., and certain nitrogen oxide compounds. If such interferences are suspected for a particular sample, the sample can be checked for suppression by running an aliquot of the sample with an As spike added. If significant suppression is found, the sample can be run by the method of standard additions.

5. Apparatus

- 5.1 Varian AA-375 double-beam Atomic Absorption Spectrophotometer with simultaneous D_2 background correction.
- 5.2 Westinghouse Model 185E Electrodeless Discharge Lamp Power Supply.
- 5.3 Varian A-25 strip chart recorder or other suitable recorder.
- 5.4 Hewlett-Packard A/D converter in the loop cable of an HP3357 Laboratory Automation System (LAS).
- 5.5 The hydride generation apparatus consists of a Technicon sampler, two Technicon proportioning pumps, a gas separator, water trap, and associated tubing. (See Figure 1).
- 5.5.1 Technicon Autoanalyzer Sampler II with 50-1/2 cam (50 samples per hour, sample 24 sec. and wash 48 sec.) and glass sampling probe.
- 5.5.2 Two Technicon Autoanalyzer Proportioning Pumps I (one for pumping reagents, the other for removal of waste liquid and pumping air into the gas stream to enhance arsenic sensitivity).
- 5.5.3 A gas separator fabricated from a 20 mm. diameter funnel with fritted disc, which was cut off 6.5 cm. above the fritted disc and fitted with a No. 4 rubber stopper with three holes drilled for 4 mm o.d. glass tubing. The stopper was cut off 1.8 cm. from the wide end.

A 1 ml. Kimax beaker is placed in the funnel.
(See Figure 2).

5.5.4 A glass water trap. (See Figure 2).

5.5.5 Tygon tubing and Pyrex glass fittings and coils of various sizes.

5.6 The silica tube furnace (Figure 3) is an 18 cm. long tube (10 mm. i.d.) with three 6 cm. long sidearms (5 mm. i.d.) on one side and two 3 cm. long sidearms (5 mm. i.d.) on the opposite side. The generated arsine enters the silica tube via the central tube, while an auxillary flow of nitrogen passes through the two transverse tubes on the ends to prevent ignition of liberated hydrogen in the light path and to prevent possible problems with room air currents. The tube is wrapped with 26 gauge asbestos insulated chromel A resistance wire and insulated with fiberglass cloth.

5.7 Superior Electric Company powerstat.

5.8 Two needle valve flow meters for nitrogen gas control.

6. Reagents

6.1 Potassium persulfate, ACS reagent, crystal.

6.2 11 N sulfuric acid.

6.3 Conc. hydrochloric acid. It is prudent to acquire sufficient conc. HCl of a single lot number for both addition to samples and for acid wash solution. Different lots of HCl may contain different background levels of As.

6.4 4 N hydrochloric acid.

6.5 0.9 percent sodium borohydride in 0.5 percent sodium hydroxide solution.

6.6 Stock arsenic solution, 1000 mg/l, Fisher Certified Atomic Absorption Standard Reference Solution, or prepared according to Standard Methods for the Examination of Water and Wastewater, 15th Edition, 307B. 3g., p. 175.

6.7 Intermediate arsenic solution, 2000 ug/l. Dilute 1 ml. of the As stock solution to 500 ml.

6.8 Working standards: Pipette 2, 3, 2, 3, and 5 ml. of the intermediate As solution into 500, 500, 250, 250, and 250 ml. volumetric flasks respectively to give concentrations of 8, 12, 16, 24, and 40 ug/l As. (All working standards

are preserved with 0.5 percent conc. HNO_3 .) After diluting to volume and thoroughly shaking, pipette 0, 5, 10, and 10 ml of the 40 ug/l As solution into 250, 200, 200, and 100 ml. volumetric flasks respectively to give 0, 1, 2, and 4 ug/l As working standards.

7. Procedure

- 7.1 Aliquots of samples are poured or pipetted into 25 x 200 mm. Pyrex (or Kimax) culture tubes to a 30 ml. mark. If a sample is predigested using method 4.1.3 of the EPA Manual or method 3050 of SW846, an aliquot of the digestate is pipetted into a culture tube and diluted to the mark. Tubes containing the standards and QC solution are also prepared. Usually three tubes are poured for the 4, 8, 12, 16, and 24 ug/l and QC solution, two tubes each for the 1 and 2 ug/l As solutions, and seven tubes for the 0 standard.
- 7.2 To the 30 ml. aliquots of samples and standards in the culture tubes are added 0.5 ml. of 11 N sulfuric acid and a scoop (0.2 g) of potassium persulfate. The tubes are tightly closed with Teflon-lined screw caps and shaken and are autoclaved at 121 degrees C for twenty minutes.
- 7.3 15 ml. of conc. hydrochloric acid is added to each tube and mixed.
- 7.4 Turn on the AA instrument, making sure that the instrument is in single-beam with gain turned down and all lamps off. Allow two minutes warmup before turning on a lamp. With the As EDL lamp installed in quadrant 3, turn on the EDL power supply and allow it to warm up two or three minutes.
- 7.5 Turn the lamp 3 control to a setting of 6, while noting a movement of the EDL wattmeter when passing from a setting of 4 to 5. Now promptly turn up the EDL power knob, while watching the wattmeter. The knob should be turned up in stages to avoid overshooting the recommended wattage for the As lamp (8 to 9 watts). Especially during the first five minutes, the power knob will have to be turned back slightly as the EDL lamp warms up to maintain the correct wattage.
- 7.6 Turn up the gain and dial the As wavelength, 193.7 nm. Final adjustments (focussing of lamp, adjustment of wavelength, and installation of the tube furnace in the light path) will be done after the lamp has warmed up for twenty to thirty minutes.
- 7.7 Plug the cord with alligator clips into the powerstat (powerstat off!), and tape the cord where the wires

separate to the side of the AA unit so the alligator clips can later be attached conveniently to the tube furnace leads.

- 7.8 Move the table supporting the hydride generation apparatus to the front of the bench with the AA unit. Connect the power cords for the pumps and sampler to the outlet on the bench.
- 7.9 Attach the tubing from the CRA gas controller to the bottom of the separator, and attach the tubing from the small Wilkerson regulator to the Dwyer flowmeter for flushing the ends of the tube furnace with inert gas.
- 7.10 Dip the takeup tubes for the wash solution and the NaBH_4 reagent into a beaker of distilled water. Stretch the pump tubing into place, lock down the chain drive, and start the two pumps. Open the nitrogen cylinder, adjust the regulator pressure to 20 psi, and turn on a low flow of N_2 with the CRA controller to prevent water from seeping through the fritted disc of the separator. The waste pump speed must be adjusted to remove all water pumped into the separator but take a minimum amount of gas.
- 7.11 Prepare the 0.9 percent NaBH_4 solution while waiting for the EDL lamp to warm up. Recipe: Into a 1 liter volumetric flask, add 10 ml. 50 percent NaOH solution into several hundred mls. of distilled water. Add 30 pellets (10/32") of NaBH_4 , and wash down the neck of the flask with distilled water. Leave space in the flask for easy shaking of the mixture, and allow the NaBH_4 to dissolve, being careful of possible gas buildup. After dissolution is complete, add distilled water to the mark and shake. For 500 ml. or 2 liters of the reagent, halve or double the recipe respectively.
- 7.12 After the EDL lamp has warmed up twenty minutes or so, turn on the recorder and adjust for a 100 percent reading with the wavelength optimized. Install the tube furnace, which is tied on an old air-acetylene burner by thick copper wire. Lower the burner, rotate, and move in and out to obtain maximum light throughput. Connect tubing from the center tube of the tube furnace to the water trap and tape it to the side of the AA unit above the cord from the powerstat. Connect tubing from the two sidearms to the Dwyer flowmeter and secure it with tape to the small shelf in front of the AA unit.
- 7.13 Adjustment of the tube furnace for maximum light throughput is a very delicate operation and considerable patience and skill is required. Sometimes movement of the tubing leading to the two sidearms under the tape is helpful in

making the adjustment. The alligator clips are supported by heavy strings taped at one end to the flame shield of the AA unit. The clips are supported in a position approximately suitable for attachment to the tube furnace leads. When the alligator clips are attached, care must be taken that a good electrical contact is made. Otherwise a hot spot might develop at the alligator clip-resistance wire interface by sparking and burn the wire in two. The lamp focussing knobs and wavelength dial are used to make the final adjustments. Hopefully the light throughput will be 80 percent or better of what it was before installation of the furnace.

- 7.14 The instrument is changed to double-beam and the background corrector (position A) is turned one.
- 7.15 The water trap is attached to its rubber stopper and clamped in place and leaned against the side of the 4 liter beaker. Usually 50 percent conc. H_2SO_4 is used in the water trap. This solution is poured into the open end of the water trap until overflowing, and the tilt of the trap is adjusted by moving the 4 liter beaker until the gas enters the water trap just above the acid solution without bubbling.
- 7.16 The powerstat is turned on and the voltage is adjusted to a setting of 65. The tube furnace will develop an orange glow in about five minutes.
- 7.17 Move the wash solution take-up tube from the distilled water beaker to the 4 N HCl wash solution. After two or three minutes, move the $NaBH_4$ take-up tube to a reagent bottle containing 0.9 percent $NaBH_4$ solution. When the 4 N HCl and $NaBH_4$ meet in the manifold, much H_2 gas is evolved and the reagent pump must be shut off for a few seconds to avoid overflowing the separator. The speed of the waste pump can be temporarily turned up. After gas has taken its place in the 40 ft. time-delay coil, the reagent pump may be left on, and the waste pump turned back to normal speed.
- 7.18 If the Beckman pH recorder is used, the 700 mv span is normally used. Since the signal is sent to the A/D converter, it is important that the voltage is positive. This can be checked with a multimeter to be sure.
- 7.19 The autosampler is turned on. Several high standards are poured and placed in the autosampler. (Usually three tubes containing 24, 16, and 12 ug/l As are poured and treated with 15 ml. conc. HCl for preliminary adjustments.) The CRA flow controller is adjusted to an optimum N_2 flow (200-300 ml/min) for good sensitivity and efficient cleanout between samples.

- 7.20 When conditions are satisfactory and the sensitivity has almost stabilized, the working As standards are arranged in the autosampler from highest to lowest and sampling is begun. The following standards are normally run: 24, 16, 12, 8, 4, 2, 1, and 0 ug/l.
- 7.21 When ready to begin acquisition of data with the LAS system, wait until the recorder pen is on the baseline between peaks and push the A/D start/stop button. The A/D status light will begin flashing at the rate of once per second. When the next peak appears on the recorder, label it and mark it as the first peak acquired by the LAS system. Repour the standards and run the first standard set.
- 7.22 Continue with the analytical run in a normal manner with the following special considerations:
- A. The autosampler must not be stopped, and the cam should never be pulled ahead by hand. This could cause peak windows to be out of phase with each other in the data reduction.
 - B. Run 0 standards on a regular basis to establish baseline points. Rerun low samples that immediately follow very high peaks.
 - C. Run full standard sets on a regular basis, especially if sensitivity changes are suspected. If a large dropoff in sensitivity occurs due to interference in a particular sample, the standards may need to be repeated two or three times in a row until the sensitivity approaches its former level.
 - D. Run an intermediate level standard and a 0 standard after approximately every fifth sample to check the sensitivity and to detect possible carry over interference.
- 7.23 Besides labelling the samples and standards on the recorder chart as to their identity, the samples must also be counted, and this count will be defined as the "reference number." Starting with the first peak that was marked as instructed in 7.21, count the peaks and label with their reference numbers all 0 standards and the first peak of each standard set. The reference number of the last peak in the run is the total number of samples plus standards in the run.
- 7.24 When all samples have been run, complete the run with a full standard set and QC sample. Run 0 standards until the baseline is established, then press the A/D converter start/stop button once. The status light will go out

after about one second and stay out while data is being processed. (Care must be taken that the start/stop button is not accidentally depressed while the A/D converter is on. This would cause loss of the raw data stored in file *RAW04.)

- 7.25 Remove the reagent take-up tubes from the NaBH_4 and HCl reagents and place them in a beaker of distilled water. Turn the powerstat off.
- 7.26 Turn the background corrector and recorder off. Turn the gain control switch to single beam. Turn the power control on the EDL power supply fully counterclockwise and immediately switch the hollow cathode knob for quadrant 3 to setting 0. Turn power to the EDL power supply and to the AA unit off.
- 7.27 Carefully loosen the clamp and disconnect the water trap from the rubber stopper. The trap is inverted to allow the acid solution to flow into the large beaker. Rinse the trap thoroughly with distilled water, wipe with a paper towel, and place in a drawer. The tubes extending through the rubber stopper are rinsed externally with distilled water, and the rubber stopper is remounted in the clamp used to support the water trap.
- 7.28 After several minutes of pumping rinse water through the reaction manifold, the rubber stopper is carefully removed from the separator. The reagent pump is left on for several minutes, and the rinse water is allowed to fall into the large beaker used for support of the water trap. The tubes extending through the stopper are rinsed with distilled water using a squeeze bottle, and the waste pump is turned off. Meanwhile the 1 ml. beaker is carefully removed from the separator, rinsed with distilled water, and placed in the drawer with the water trap. The separator is thoroughly rinsed by spraying inside with distilled water, and then it is supported in inverted position by its clamp while nitrogen gas is allowed to continue flowing for several minutes. (The separator must be disassembled in this manner after each use, and water must not be allowed to sit in contact with the fritted disc. This can cause loss of porosity of the fritted disc.)
- 7.29 After several minutes the reagent pump is turned off. For both pumps, the roller head is unlatched and placed in its upright position, and the reagent line block is disconnected to loosen the reagent lines. The nitrogen supply is shut off at the tank.

8. Data Reduction

- 8.1 In case of computer failure, the peaks obtained on the recorder may be measured manually and used for quantitation of the samples. A calibration curve or curves are constructed from the standards peak height data taking into account changes in sensitivity during the run. The calibration points are fitted by the second order least squares method forced through the origin, and sample concentrations are calculated from peak heights using the calculated coefficients of the quadratic equation.
- 8.2 For data reduction using the HP3357 LAS system, a FORTRAN program called HYDIV, a transfer file called TES21, and a BASIC program called HYDFIN are used. The running of program HYDIV should be done on the HP 2623A Graphics Terminal. Transfer file TES21 can be run on any video terminal connected to the HP3357 system. It is preferable to run program HYDFIN on a video terminal to check the results and see if the proper baseline points have been chosen, but the final hard copy run of HYDFIN can be carried out on the TI Model 733 teletype terminal.
- 8.3 To run program HYDIV, log on to the HP 2623A terminal according to the procedure specified by the system manager. In RTE mode immediately after the colon cursor, type HYDIV. Program HYDIV requests the name of the file to be analyzed. For the default file associated with A/D channel 4, type *RAW04. Program HYDIV searches for peaks using the derivative of the analytical data and lists the slice numbers of peaks found. After the data for the entire run has been searched, the slice numbers of the peaks found are fitted by linear regression versus sample numbers. The slope should be approximately 144 or twice the sampling rate of 72 seconds since a reading is acquired every 1/2 second. The Y-intercept corresponds to the theoretical slice number of sample number 0. Using the coefficients of the regression equation, a map file is calculated giving the slice number of all peaks in the run.
- 8.4 Program HYDIV then gives the user the option of going to the graphics mode to check the integration limits for suitability to the particular data. After display of the first 13 peaks of the run, the user may use the soft keys to change the integration limits. The next screen may be viewed, or advancement to a later stage in the run may be expedited. When the user is satisfied with the integration width chosen, he exits the graphics mode. Program HYDIV automatically integrates the analytical data using the chosen integration limits and the peak positions from the map file. The peak areas are stored in file HARRAY.

- 8.5 The data in file HARRAY must be converted from type 4 to type 69 in order for it to be accessible by BASIC program HYDFIN. To carry out this conversion, simply type TR, TES21, HARRAY after the colon cursor. During the conversion process, the user needs only to obey the prompt: RUN HY69::21, then type BYE. The data from file HARRAY will then be in file HYD69 as type 69 data.
- 8.6 To enter BASIC mode from RTE, type BSC57. Type RUN HYDFIN. This loads program HYDFIN into the BASIC program work area of the computer and begins running it. To the prompt "Analysis of ?", type AS. To the prompt "Enter the raw file name?", type the raw file name which will in most cases be the default file *RAW04.
- 8.7 To abort a BASIC program at a prompt, type Q with the CONTROL key held down and then hit RETURN. To abort while a program is processing, hit a key to get into break mode. Type BR. This places the computer in BASIC break mode. Now type ABORT to get back into BASIC.
- 8.8 After the computer prints several lines of header information, the prompt "List calculated peaks?" appears. Usually it is best to respond yes for the final hard copy to have complete documentation of integrated peaks obtained in the run.
- 8.9 The next prompt requests the peak number of the first peak in 3 peak sets for carry over calculation. Enter the numbers listed on the sheet of paper that were taken from the recorder tracing of the run. The computer will calculate the percent carry over for each set entered. Enter 0 as the last number. Now the computer gives the user the opportunity to enter the estimated percent carry over. This can be the same as the average percent carry over, which the computer just calculated, or it can be another figure that the user selects. (Usually the figure is between 0.5 and 2. The effect of carry over is minimal for most samples, but it can be significant where a sample at relatively high analyte level just precedes a sample of very low concentration.)
- 8.10 The next prompt requests the reference number of a peak after which an abrupt baseline shift occurred. The most common cause of such a shift is turning off or on the background corrector. This feature is used to cause extrapolation of baseline to the baseline shift point and not attempt to establish the baseline as the direct line between adjacent baseline points on each side of the abrupt shift.
- 8.11 The next prompt requests the reference number of peaks that may be considered as baseline. The choice of

baseline points is up to the discretion of the operator who views the recorder tracing. If a very high standard or sample just precedes a zero standard, that zero standard may possibly not be a good choice for a baseline point because of carry over. The computer establishes its baseline by calculating the straight line between adjacent baseline points. The carry over correction is performed prior to the computation of baseline and the subtraction of all the integrated area below the baseline.

- 8.12 The next prompt gives the option to have the calculated peaks listed. (Usually this listing of the peaks is omitted in the final hard copy.)
- 8.13 The computer is now ready to calculate the standard curves for the individual standard sets of the run. After the prompt, enter the reference number of the first standard in a standard set. (The standard concentrations of the standard set must correspond to the values given in 7.20 above and be in the same order.) The next prompt gives the option of dropping one of the standards and its associated area from the computation if, for some reason, it was of incorrect concentration or otherwise not of appropriate response. If this option is not desired, enter 99. The computer now uses the method of second order least squares and second order least squares through the origin to calculate the coefficients of the quadratic model equations of the standard curves. The coefficients are printed along with the standard error of estimate for each of the curve fits. Usually the standard error of estimate for As data will be in the range from 10^{10} to 10^{12} . When higher values of the standard error of estimate are noted, the peak heights of the samples on the recorder should be checked to see if one of the standards is out of line.
- 8.14 After computation of the calibration coefficients for the standard sets, enter 0 at the prompt, and the computer will begin printing out the sample results. The concentrations of the individual standards in the standard sets are printed by the computer. For all other standards and samples, a space is left for the operator to enter the appropriate sample designation. Although results are given for two separate types of computation, the results will usually be very similar, and the results for the calibration by second order least squares forced through the origin will normally be reported. On the CRT terminal, hit a key periodically to stop scrolling of the data and check the results to see if they look appropriate. Hit the RETURN key to continue scrolling.
- 8.15 After viewing the data on a CRT terminal, move to a teletype terminal, log on, and repeat the calculations to get a hard copy. At present, the results are transferred

by hand to bench sheets for preparation of reports and designation of reported values.

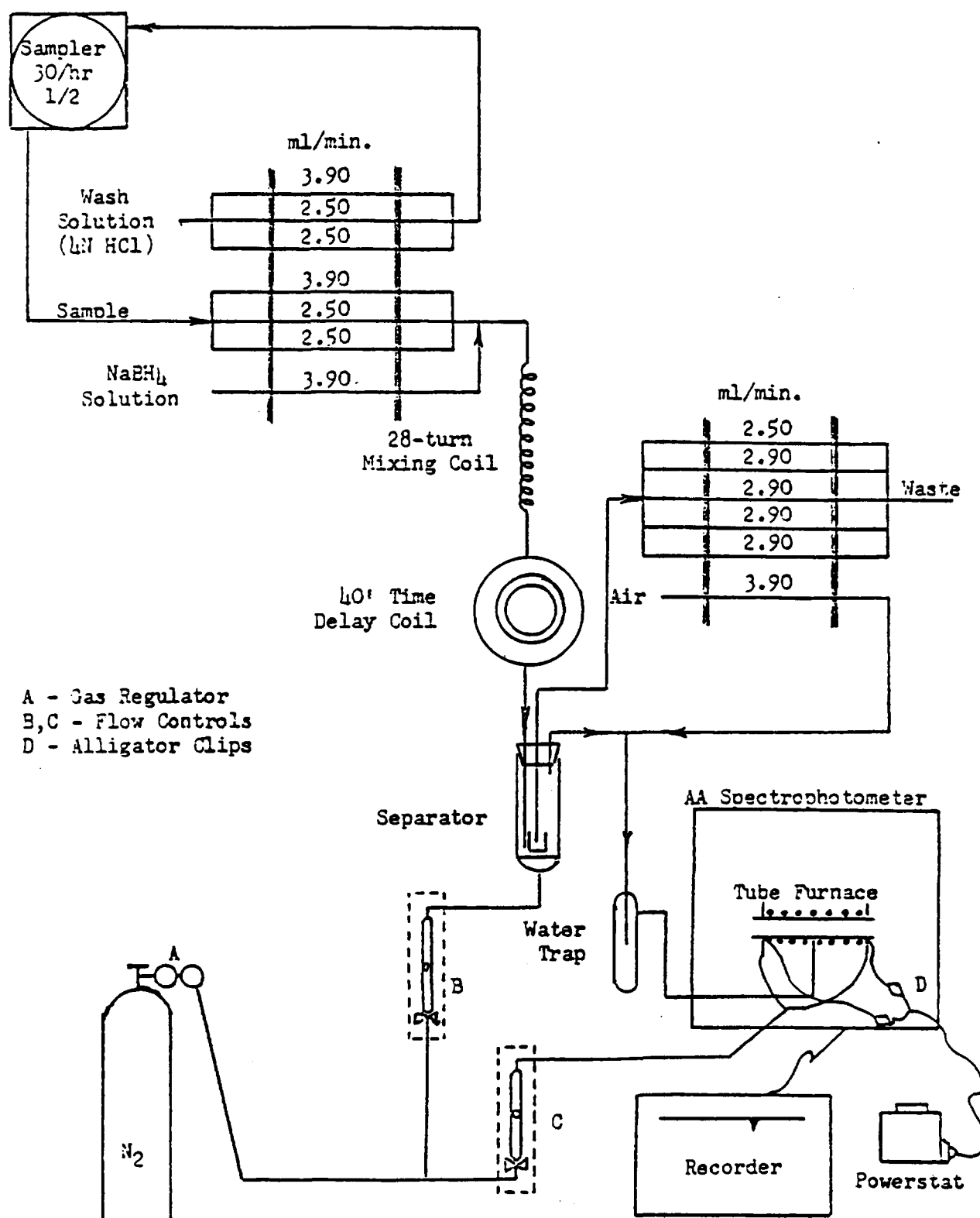
9. Precision and Accuracy

- 9.1 A Quality Control sample is prepared with each As run and determined at least once for every 20 samples.
- 9.2 Duplicate samples are run at the rate of at least one for every 20 samples.
- 9.3 If possible interferences are suspected for certain samples, spiked samples are prepared to check for suppression.
- 9.4 The method of standard additions is used to quantitate samples for which the degree of suppression is significant.
- 9.5 EPA Quality Control Samples (SRM) and various performance evaluation samples are determined periodically.

10. References

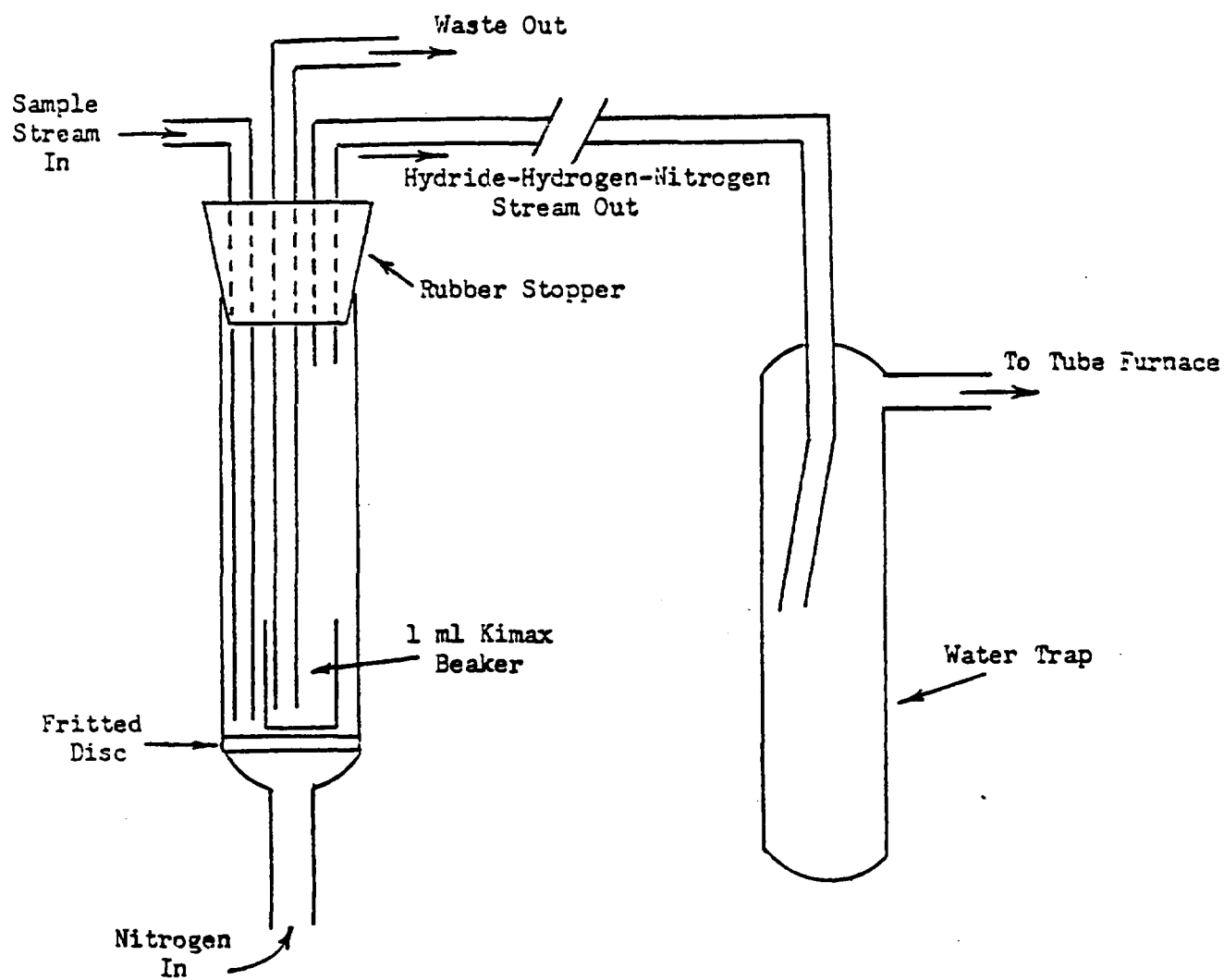
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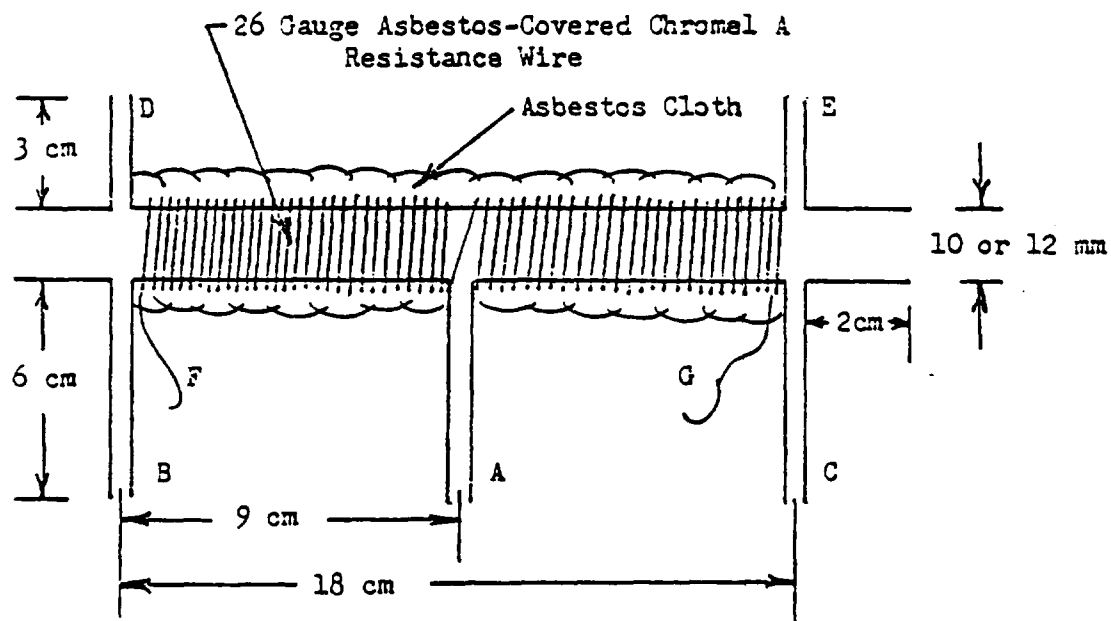
SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR AUTOMATED ARSENIC DETERMINATION

FIGURE 1



GAS SEPARATOR AND WATER TRAP

FIGURE 2



- A - Central sidearm for introduction of gaseous hydride (5 mm i.d.)
 B,C,D,E - Transverse tubes for auxiliary N₂ flow (5 mm i.d.)
 F,G - Resistance wire leads

Electrically Heated Silica Tube Furnace

5-28-80
DGR, RAS

Recovery Study: Arsenic (III) and Organic Arsenic

Distilled water, well water (obtained at DGR's home on 4-9-80), and river water (obtained from the White River at the Indianapolis Water Company on 4-15-80) were spiked with arsenic (III), cacodylic acid, and p-arsanilic acid at approximately the 4 ug/l level. The stock solutions of cacodylic acid and p-arsanilic acid were diluted and analyzed with respect to arsenic (III) oxide standards by direct flame AA using a nitrous oxide/acetylene flame. The cacodylic acid was calculated to be 3.85 ug/l and the p-arsanilic acid was 3.91 ug/l in the spiked solutions. All the spiked and unspiked samples were acidified with conc. nitric acid to give 0.5% acid solution.

Routine analytical tests were performed on the well water and river water used, and the results are given in ATTACHMENT C.

Seven aliquots from each of the twelve solutions prepared were poured and carried through the digestion and analysis procedure. The results are given on the following pages.

An aliquot of each of the twelve samples was treated with the appropriate quantity of hydrochloric acid and analyzed directly by the automated procedure without digestion. The results are given below:

<u>Sample</u>	<u>ug/l AS</u>	<u>% Recovery</u>
D -- Distilled water, as received	<0.1	---
D3 - Distilled water plus 4.0 ug/l As(III)	3.7	92.5%
DC - Distilled water plus 3.85 ug/l org. As (cacodylic acid)	0.4	10.4%
DP - Distilled water plus 3.91 ug/l org. As (p-arsanilic acid)	0.3	7.8%
W -- Well water, as received	4.6	---
W3 - Well water plus 4.0 ug/l As(III)	8.3	92.5%
WC - Well water plus 3.85 ug/l org. As (cacodylic acid)	5.0	10.4%
WP - Well water plus 3.91 ug/l org. As (p-arsanilic acid)	5.0	10.2%
R -- River water, as received	0.8	---
R3 - River water plus 4.0 ug/l As(III)	4.6	95.0%
RC - River water plus 3.85 ug/l org. As (cacodylic acid)	1.3	13.0%
RP - River water plus 3.91 ug/l org. As (p-arsanilic acid)	1.3	12.8%

PRECISION & ACCURACY DATA

ISBH Code No. _____
Storet No. _____

Determination: ARSENIC

Sample Source: Distilled Water

Chemist: DGR, RAS

Date: 5/28/80

Runs	D	Sample	D3
	As Rec'd.	<u>4.0</u> ug/l Added	
1	<0.1		4.0
2	<0.1		4.0
3	<0.1		4.0
4	<0.1		4.0
5	<0.1		4.0
6	<0.1		4.0
7	<0.1		3.9

Mean: <0.1 3.99

s: -- + 0.04

C.V.: -- 0.9 %

% Recovery: 99.8 %

Relative Error: 0.2 %

Remarks:

METHOD: Preliminary digestion by persulfate-autoclave procedure. Addition of hydrochloric acid and automated arsine generation using sodium borohydride reagent. Detection by AA using an electrically heated tube furnace and data output by recorder.

Reference: ISBH Water & Sewage Laboratory Division Procedure, 1980

PRECISION & ACCURACY DATA

ISBH Code No. _____

Storet No. _____

Determination: ARSENICSample Source: Distilled WaterChemist: DCR, RASDate: 5/28/80

Runs	Sample	
	DC	DP
	<u>3.85 ug/l Added</u>	<u>3.91 ug/l Added</u>
1	3.9	3.9
2	3.7	4.0
3	3.7	4.0
4	3.7	4.0
5	3.8	4.0
6	3.9	3.9
7	3.8	3.9

Mean: 3.79 3.96s: ± 0.09 ± 0.05C.V.: 2.4 % 1.4 %% Recovery: 98.4 % 101.3 %Relative Error: 1.6 % 1.3 %

Remarks:

ibid.

PRECISION & ACCURACY DATA

ISBH Code No. _____
Storet No. _____

Determination: ARSENIC

Sample Source: Well Water

Chemist: DJR, RAS

Date: 5/28/80

Runs	W	Sample	W3
	As Rec'd.	<u>4.0</u> ug/l Added	
1	5.0		8.9
2	5.0		9.3
3	5.1		9.2
4	4.9		8.5
5	5.0		8.9
6	5.0		8.9
7	5.0		9.0

Mean: 5.00 8.96

s: ± 0.06 ± 0.26

C.V.: 1.2 % 2.9 %

% Recovery: 99.0 %

Relative Error: 1.0 %

Remarks: ibid.

PRECISION & ACCURACY DATA

ISBH Code No. _____
Storet No. _____

Determination: ARSENIC

Sample Source: Well Water

Chemist: DJR. RAS

Date: 5/28/80

Runs	WC	Sample	WP
	<u>3.85 ug/l Added</u>		<u>3.91 ug/l Added</u>
1	8.8		8.8
2	9.1		8.8
3	8.9		9.0
4	8.9		8.8
5	8.8		9.0
6	9.0		9.0
7	8.6		8.8

Mean:	<u>8.87</u>	<u>8.89</u>
s:	<u>± 0.16</u>	<u>± 0.11</u>
C.V.:	<u>1.8 %</u>	<u>1.2 %</u>
% Recovery:	<u>100.5 %</u>	<u>99.5 %</u>
Relative Error:	<u>0.5 %</u>	<u>0.5 %</u>

Remarks: ibid.

PRECISION & ACCURACY DATA

ISBH Code No. _____
Storet No. _____

Determination: ARSENIC

Sample Source: White River Water

Chemist: DCR, RAS

Date: 5/28/80

Runs	R	Sample	R3
	As Rec'd.	<u>4.0</u> ug/l Added	
1	1.3		5.4
2	1.3		5.3
3	1.3		5.5
4	1.3		5.4
5	1.3		5.2
6	1.3		4.9
7	1.3		5.1

Mean: 1.30 5.26

s: -- ± 0.21

C.V.: -- 3.9 %

% Recovery: 99.0 %

Relative Error: 1.0 %

Remarks: ibid.

PRECISION & ACCURACY DATA

ISBH Code No. _____
Storet No. _____

Determination: ARSENIC

Sample Source: White River Water

Chemist: DGR, RAS

Date: 5/28/80

Runs	Sample	
	RC	RP
	<u>3.85 ug/l Added</u>	<u>3.91 ug/l Added</u>
1	4.8	5.0
2	4.5	5.1
3	4.9	5.0
4	4.9	5.1
5	5.0	5.2
6	4.9	5.1
7	5.0	5.1

Mean:	<u>4.86</u>	<u>5.09</u>
s:	<u>± 0.17</u>	<u>± 0.07</u>
C.V.:	<u>3.5 %</u>	<u>1.4 %</u>
% Recovery:	<u>92.5 %</u>	<u>96.9 %</u>
Relative Error:	<u>7.5 %</u>	<u>3.1 %</u>

Remarks: ibid.

Recovery Study: EPA Quality Control Samples (SRM). For the results of routine analytical tests performed on the river water used in this study, see ATTACHMENT C.

Sample	As Spike Value (ug/l)	Arsenic Found		% Recovery	
		In Distilled Water (ug/l)	In River Water ug/l)	In Distilled Water	In River Water
1	26.	24.5	24.4	94.2%	88.8%
2	109.	101.	105.	92.7%	95.1%
3	154.	144.	143.	93.5%	92.0%
Distilled		< 0.1			
River			1.3		

ATTACHMENT C

Analytical data for the well water obtained at DGR's home on 4-9-80 and the river water obtained from the White River at the Indianapolis Water Co. on 4-15-80 are given below. The well water was used in the study described in paragraph 9.1, and the river water was used in the studies described in paragraphs 9.1 and 9.2.

	Well Water (mg/l)	River Water (mg/l)
pH	7.3	7.75
Sp. Cond. (umhos/cm)	---	536.
Total Solids	---	400.
Total Dissolved Solids	320.	350.
Suspended Solids	---	44.
Hardness as CaCO ₃	280.	262.
Calcium	67.	72.
Magnesium	27.	20.
Sodium	40.	12.
Potassium	1.8	2.5
MO Alk. as CaCO ₃	376.	184.
Chloride	7.	27.
Sulfate	< 5.	46.
Phosphate	0.3	---
Fluoride	0.8	0.22
Nitrate	< 0.1	5.0
Barium	0.22	---
Iron	1.58	0.49
Manganese	0.03	0.04

COMPARISON OF AUTOMATED AND MANUAL
DETERMINATION OF ARSENIC (All results in ug/l)

Lab No.	Source or Sample Type	Dil'n.	Automated Hydride Generation	Manual Hydride Generation
CO356	Bloomfield Water Dept. (Tubular Wells - Sand)		0.4	< 0.5
CO368	Waveland Water Works (Tubular Wells - Limestone)		0.6	0.5
CO615	Scottsburg Water Dept. (Impoundment)		0.1	< 0.5
DO112	Vermillion River - 0.8		0.8	0.8
DO121	Wildcat Creek - 69		0.4	< 0.5
DO122	Wildcat Creek - 63		1.1	0.9
DO126	Outfall, Jasper	1/100	610.	610.
DO127	Stream, Jasper	1/100	190.	250.
"	" "	1/20	197.	158., 197.
DO135	Princeton Sewage Treatment Plant - Raw		1.4	1.1, 0.8
DO136	Princeton Sewage Treatment Plant - Final		0.9	0.7
DO137	Outfall, Princeton	1/20	2.	< 10.
"	" "	25/30	1.7	2.8
"	" "	1/1	1.8	2.0
DO138	Outfall, Princeton		1.9	1.0
DO139	Outfall, Princeton		1.0	1.4
DO140	Outfall, Princeton		0.9	1.1
DO148	Eagle Creek - 21		0.4	< 0.5
DO149	Eagle Creek - 1		0.8	0.9
DO239	Kankakee River - 65	25/30	1.6	1.7
"	" "	1/1	1.6	1.6
DO243	Lake Michigan - Whiting		1.1	0.5
DO252	Burns Ditch - 0		1.0	0.8, 0.5

COMPARISON OF AUTOMATED AND MANUAL
DETERMINATION OF ARSENIC (All results in ug/l)

Lab No.	Source or Sample Type	Dil'n.	Automated Hydride Generation	Manual Hydride Generation
DO255	Kankakee River - 125		1.3	1.1
DO256	St. Joseph River - 46		0.9	0.9
DO258	St. Joseph River - 78		1.0	1.0
DO260	Kankakee River - 65		1.7	1.1
DO438	Alcoa, Newburg (Fly ash pit)		16.8	15.0, 16.8
DO440	Alcoa, Newburg (Sampling station)		1.7	2.0
DO441	Alcoa, Newburg (Pipe discharge)		2.6	2.2
DO442	Alcoa, Newburg (Cooling tower blowdown)		6.9	7.7, 6.0
CI659	Plymouth Water Dept. (Gravel packed wells)		0.7	0.9
CI660	Frankfort Water Works (Tubular & gravel packed wells)		0.4	< 0.5
CI827	New Castle State Hospital (New well)		5.3	6.4
CI964	Mounds State Park, Anderson (Artesian well)		0.6	0.7
DO515	Outfall, Avon		10.2	11.5, 10.7
DO529	Vermillion River - 0.8		1.5	1.4
DO544	Wildcat Creek - 69		1.0	1.2
DO545	Wildcat Creek - 63		1.8	2.1
DO547	Wildcat Creek - 63		1.8	1.9
DO553	Outfall, Greensburg		0.1	< 0.5
DO594	Kankakee River - 65		2.3	2.7
DO599	Lake Michigan - Whiting		0.8	0.9
DO600	Indiana Harbor Canal - 0		1.5	1.5
DO620	Burns Ditch - 0		1.5	1.5, 2.0
DO623	Trail Creek - 0.3		0.8	1.0

COMPARISON OF AUTOMATED AND MANUAL
DETERMINATION OF ARSENIC (All results in ug/l)

Lab No.	Source or Sample Type	Dil'n.	Automated Hydride Generation	Manual Hydride Generation
DO624	Lake Michigan - Michigan City		1.0	1.5
DO626	Kankakee River - 125		6.4	8.3
DO631	St. Joseph River - 46		1.1	1.4
DO633	St. Joseph River - 78		1.6	1.6
DO635	St. Joseph River - 78		0.9	1.1
DO641	Well, Warsaw		4.2	4.7
DO642	Well, Warsaw		1.7	1.8
DO630	Plating Plant Outfall, Dillsboro	1/10	2.	< 5.
"	" " "	1/3	1.9	3.8
"	" " "	25/30	2.0	2.9
C2070	Ft. Wayne Water Dept. (St. Joseph River)		0.3	0.7
C2071	Bluffton Water Dept. (Wells - limestone & Wabash R.)		0.3	< 0.5
C0272	Montpelier Water Dept. (Salamonie River)		0.1, <0.1	< 0.5
C2073	Hentington Water Dept. (Wells-limestone & Wabash R.)		0.4	0.5
C2075	Elkart Water Works (Gravel packed wells)		3.1	2.7
C2162	Turkey Run State Park, Marshall (Spring)		0.1	1.1
C2163	Jasper-Paluski Fish & Wildlife Area, Medaryville (Well)		1.1	1.6
C2164	Jasper-Paluski State Nursery, Medaryville (Well)		0.7	1.0
C2165	Kankakee State Fish & Wildlife Area, Knox (Well)		< 0.1	< 0.5
C2345	Muncie Water Dept. (White R., impoundment, & Tubular Wells)		0.4	< 0.5
C2426	Ramsey Water Co. (Tubular wells - limestone)		0.2	< 0.5
C2429	Hoosier Water Co., Brownstown (Tubular wells - gravel)		0.4	0.5
C2432	Morristown Water Dept. (Tubular wells - gravel)		1.4	1.8

COMPARISON OF AUTOMATED AND MANUAL
DETERMINATION OF ARSENIC (All results in ug/l)

Lab No.	Source or Sample Type	Dil'n.	Automated Hydride Generation	Manual Hydride Generation
C2492	Fawn River State Fish Hatchery, Orland (Well)		1.6, 1.5	0.9, 2.0
C2493	Curtis Creek Trout Rearing Station, Howe (Well)		0.1	< 0.5
C2494	Tri Lake Fisheries Station, Columbia City (Well)		1.5	1.4
C2495	Tri Lake Fisheries Station, Columbia City (Well)		1.0	1.2
C2497	Darlington Water Co. (Well #1)		0.3	< 0.5
C2498	Darlington Water Co. (Well #2)		0.1	< 0.5
A0030	Greenwood Springs Drinking Water Ft. Wayne (Bottled Water)		< 0.1	< 0.5
A0031	Greenwood Springs Demineralized Water, Ft. Wayne		< 0.1	< 0.5
A0032	Gackenhimer Pharmacy Distilled Water, Wabash		< 0.1	< 0.5
DC659	Eagle Creek - 1		1.1	1.0
DC660	Eagle Creek - 21		1.4	1.4
DO721	Lake, Goodnight Farm, Frankfort		1.9	1.4
DO722	Well, Goodnight Farm, Frankfort		0.3	< 0.5
DO731	Well, Seymour		6.1	6.7
DO778	Lake, Midwest Steel, Portage		1.0	1.4
DO780	Outfall, Midwest Steel, Portage		0.5	0.5
DO803	Runoff, Ligonier		0.7	0.5
DO804	Runoff, Ligonier		0.7	0.8
DO806	Runoff, Ligonier		0.9	< 0.5
DO807	Runoff, Ligonier		0.7	0.9
DO909	Eagle Creek - 21		0.7	< 0.5
DO910	Eagle Creek - 1		1.1	1.1
DO923	Ditch, Enviro Chem, Boone County		3.9	3.0

COMPARISON OF AUTOMATED AND MANUAL
DETERMINATION OF ARSENIC (All results in ug/l)

Lab No.	Source or Sample Type	Dil'n.	Automated Hydride Generation	Manual Hydride Generation
D0924	Ditch, Enviro Chem, Boone County		0.6	0.7
D0927	Creek, Enviro Chem, Boone County		0.7	0.6
C2610	Purdue University, Lafayette (Wells)		1.9	2.0, 2.1
C2677	Shrader Weaver Nature Preserve, Connersville (Well)		2.7	1.9
C2810	Lawrenceburg Water Works (Tubular wells - sand)		0.3	< 0.5
C2811	Vevay Water Dept. (Tubular wells - gravel)		0.1	< 0.5
C2812	Well, Elrod Residence		1.0	0.9
C2816	Batesville Water Utility (Impounding reservoirs)		0.6	< 0.5
C2819	Osgood Water Dept. (Laughery Creek)		0.4	< 0.5
C2825	Covington Light & Water Utility (Gravel packed wells)		0.2	< 0.5
C3020	Ind. Cities Water Corp., Jeffersonville (Tubular & gravel packed wells)		0.7	1.2, 0.7
C3024	Ind. Cities Water Corp. New Albany, (Ohio River)		0.2	< 0.5
C3071	Brookville Reservoir, Brookville (Well)		0.2	< 0.5
C3072	Brookville Reservoir, Brookville (Well)		0.1	< 0.5
C3025	Water Utilities Inc., Warsaw (Center Lake)		0.6	< 0.5
C3214	Water Utilities Inc., Warsaw (Gravel packed wells)		0.5	< 0.5
C3217	Water Utilities Inc., Warsaw (Gravel packed wells)		0.4	0.5
C3219	American Water Works, Richmond (Whitewater River, wells & springs)		0.5	< 0.5
C3443	Riley Water Works (Tubular wells - rock)		0.7	< 0.5
C3444	Glendale Hatchery, Montgomery (Well)		8.5	7.5
D0730	Well, Seymour	1/4	29.6	37.
D0999	Outfall, Grissom AFB, Peru		3.2	3.5, 3.4
D1000	Well, Grissom AFB, Peru		4.5	4.6

COMPARISON OF AUTOMATED AND MANUAL
DETERMINATION OF ARSENIC (All results in ug/l)

Lab No.	Source or Sample Type	Dil'n.	Automated Hydride Generation	Manual Hydride Generation
D1001	Well, Grissom AFB, Peru		11.8	12.2
D1002	Well, Grissom AFB, Peru		3.3	3.6
D1011	Vermillion River - 0.8		0.9	< 0.5
D1029	Wildcat Creek - 69		0.9	0.9
D1030	Wildcat Creek - 63		4.1	3.8
D1055	Kankakee River - 65		2.2	2.4
D1060	Lake Michigan - Whiting		0.9	1.0
D1061	Indiana Harbor Canal - 0		1.9	1.2, 1.9
D1083	Burns Ditch - 0		1.3	0.8
D1086	Trail Creek - 0.3		1.0	1.3
D1087	Lake Michigan - Michigan City		0.9	0.9
D1089	Kankakee River - 125		1.8	2.1
D1100	St. Joseph River - 46		1.4	1.6
D1102	St. Joseph River - 78		1.5	1.5
D1104	St. Joseph River - 78		1.3	1.3
D1247	Eagle Creek - 1		1.9	1.7
D1272	Eagle Creek - 21		1.4	1.0
C3277	Bass Lake State Beach, Knox (Well)		12.0	9.7, 13.8
C3278	Bass Lake Hatchery, Knox (Well)		3.4	3.7
D1172	Outfall, Vincennes	1/10	114.	129.
D1174	Creek, Vincennes	1/10	58.	61.
D0449L	Sludge leachate, Huntingburg	1/30	< 3.	< 15.
"	"	1/10	< 1.	< 5.
"	"	25/30	< 0.1	< 0.5

Method Comparison and Recovery Study: Selected samples were spiked with two levels of arsenic and analyzed by both the automated and manual methods. The arsenic found results are determined values uncorrected for dilution factors, and the % recovery results are calculated by the formula

$$\frac{(\text{As found in spiked samples})}{(\text{As spike}) + (\text{As found in unspiked sample})} \times 100\%$$

The sample source and concentration of possible interferences, where available, are listed.

	Dilution	As Added (ug/l)	Automated Method		Manual Method	
			As found (ug/l)	% Recovery	As found (ug/l)	% Recovery
DO239 (Kankakee River - 65, COD 14. mg/l, Cu 0.02 mg/l)	25/30	0.0 5.33 10.67	1.3 6.9 11.7	--- 104. 98.	1.4 6.4 13.3	--- 95. 110.
DO137 (Outfall, Princeton, COD 1300 mg/l, Cu 41. mg/l, Ni 0.48 mg/l)	25/30	0.0 5.33 10.67	1.4 4.1 ---	--- 61. ---	2.3 7.2 9.2	--- 94. 71.
DO553 (Outfall, Greensburg, Cu 0.36 mg/l, Ni 0.06 mg/l)	25/30	0.0 5.33 10.67	0.1 5.2 9.3	--- 96. 86.	< 0.5 6.7 13.1	--- 126. 123.
DO630 (Outfall, plating plant, Dillsboro, Cu 0.50 mg/l, Ni 52. mg/l)	1/10	0.0 5.33 10.67	0.2 5.5 10.6	--- 99. 98.	< 0.5 5.5 9.7	--- 103. 91.
	1/3	0.0 5.33 10.67	0.6 5.5 10.4	--- 93. 92.	1.3 7.3 11.7	--- 110. 98.
	25/30	0.0 5.33 10.67	1.7 5.8 10.3	--- 83. 83.	2.4 5.5 11.8	--- 71. 90.
DO449L (Sludge leachate, Huntingburg, Cu 180. mg/l, Ni 28. mg/l)	1/30	0.0 5.33 10.67	< 0.1 5.0 10.1	--- 94. 95.	< 0.5 5.8 11.2	--- 109. 105.
	1/10	0.0 5.33 10.67	< 0.1 3.2 7.2	--- 60. 67.	< 0.5 4.9 9.2	--- 92. 86.
	25/30	0.0 5.33 10.67	< 0.1 1.9 3.2	--- 36. 30.	< 0.5 3.7 9.8	--- 69. 92.

BIOCHEMICAL OXYGEN DEMAND (BOD)
5 Days, 20° C.

ISBH Code No. BOD-2-88
STORET No. 00310
Approved for NPDES

1. Scope and Application

- 1.1 The biochemical oxygen demand is used for determining the relative oxygen requirements of municipal wastewater, industrial wastes, and surface waters.
- 1.2 The limit of detection is 1 mg/l and the working range is 1 to 8 mg/l.

2. Summary of Method

- 2.1 The BOD test is an empirical bioassay-type procedure which measures the dissolved oxygen consumed by microbial life while assimilating and oxidizing the organic matter present. The standard test conditions include dark incubation at 20° C. for a five-day period. The reduction in dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand.

3. Sample Handling and Preservation

- 3.1 A two-quart polyethylene bottle is an acceptable container.
- 3.2 All samples must be cooled to 4° C. until such time as the dilutions are prepared for the BOD determination.
- 3.3 BOD dilutions should be prepared and incubation started within 48 hours after the sample has been collected or the compositing has been completed.

4. Comments

- 4.1 Samples should be warmed to 20° C. before analysis.
- 4.2 The pH of the sample should be between 6 and 8.
- 4.3 Residual chlorine should be removed before analysis.
- 4.4 Any sample with dissolved oxygen concentration of 9.0 mg/l or more at 20° C. is considered supersaturated and must be corrected before dilutions are made.
- 4.5 Some types of wastes (high in metals, cyanide, pesticide, or herbicide wastes) may be toxic to the microorganisms used to seed the sample dilutions. If toxicity is suspected, it should be recorded on the laboratory bench sheet, and

the final report form for the sample. To obtain valid BOD results on this type of waste, the seed material used to prepare sample dilutions must be acclimated to the waste.

5. Apparatus

- 5.1 BOD incubator which will maintain a temperature of $20 \pm 1^\circ \text{C}$. and also exclude light.
- 5.2 YSI Model 54 Oxygen Meter, or the equivalent, dissolved oxygen probe and standard membrane kit.
- 5.3 Magnetic stirrer.

6. Reagents

6.1 Dissolved Oxygen Determination

- 6.1.1 Distilled water which is free of chlorine residual.
- 6.1.2 Manganese sulfate solution: Dissolve 364 g mangamous sulfate monohydrate in 700 ml distilled water. Dilute to one liter and filter before use. This solution should not give a color with starch when added to an acidified solution of potassium iodide.
- 6.1.3 Alkali-iodide-azide-reagent: Add 600 g reagent grade potassium iodide (KI), 1000 ml of distilled water, 2600 ml 50 percent NaOH and 40 g NaN_3 . Dilute to 4 liters with distilled water. Store in a polyethylene bottle with a tight fitting cap. (Warning! This reagent is extremely caustic and may cause serious burns if splashed on skin or eyes. Sodium azide will form explosive azides with lead or copper plumbing and should be used only with plastic or glass drains and pipes.)
- 6.1.4 Sulfuric acid, concentrated, reagent grade.
- 6.1.5 Sulfuric acid solution, (1 + 9): Add 10 ml reagent grade sulfuric acid to 90 ml distilled water. Mix and cool to room temperature before use.
- 6.1.6 Starch solution: Dissolve 16 g zinc chloride in 800 ml boiling distilled water. Add a cold water suspension of 20 g soluble potato starch and stir. After two minutes add 200 ml distilled water, boil for two minutes more, cool, and allow to settle overnight.
- 6.1.7 Potassium iodide, crystal or granular, reagent grade, iodate free.

- 6.1.8 Potassium dichromate solution, 0.0250 N: Dry primary standard grade potassium dichromate at 103° for two hours, then dessicate at room temperature for one hour. Dissolve 1.226 g potassium dichromate in 500 ml distilled water and dilute to one liter. Store in a tightly capped bottle. Prepare fresh monthly.
- 6.1.9 Sodium thiosulfate titrant, approximately 0.0350 N: Add 8.6863 g of sodium thiosulfate pentahydrate to 500 ml distilled water, add 1.5 ml 6 N sodium hydroxide, and dilute to one liter. Allow this solution to remain undisturbed for 24 hours before standardization. The procedure for standardization follows:
- Dissolve approximately 2 g potassium iodide crystals in 150 ml distilled water. Add one mL of 50 percent sulfuric acid and then 20.00 ml potassium dichromate solution. Dilute to 100 ml, mix, and place the titration vessel in the dark for 5 minutes before titrating.
 - Titrate the solution prepared above with the sodium thiosulfate titrant until a pale straw color is reached. Add approximately one ml starch solution and continue the titration until the blue color just disappears. Record the volume of sodium thiosulfate titrant used.
 - Repeat steps a & b at least three times until three titrations match within 0.10 ml. Average three values.
 - Calculate the normality of the sodium thiosulfate solution:

$$N = \frac{(0.025 \text{ N.} \times 20 \text{ ml})}{\text{average value from step c}}$$

Standardize the titrant each week it is to be used. The date, analyst name, normality of the thiosulfate should be recorded on each BOD work sheet.

6.2 BOD Determination

- 6.2.1 Acid solution, 1 N: Dissolve 28 ml reagent grade concentrated sulfuric acid in 500 ml distilled water. Cool to room temperature and dilute to one liter with distilled water.

- 6.2.2 Base solution, 1 N: Dissolve 40 g reagent grade sodium hydroxide in 500 ml distilled water. Cool to room temperature and dilute to one liter with distilled water.
 - 6.2.3 Calcium chloride solution: Dissolve 27.5 g anhydrous reagent grade calcium chloride in distilled water and dilute to one liter.
 - 6.2.4 Distilled water: Store sufficient chlorine-free distilled water in a loosely-capped, chemically clean, glass or plastic carboy (2.5 or 5 gal) in the 20° C. incubator. The storage period should be no less than one week, after the nutrients have been added, unused water should be dumped after 48 hours and replaced with fresh.
 - 6.2.5 Ferric chloride solution: Dissolve 0.25 g reagent grade ferric chloride hexahydrate, in 500 ml distilled water. Dilute to one liter with distilled water.
 - 6.2.6 Magnesium sulfate solution: Dissolve 22.5 g reagent grade magnesium sulfate, heptahydrate, in 500 ml distilled water. Dilute to one liter with distilled water.
 - 6.2.7 Phosphate buffer solution: Dissolve 8.5 g reagent grade potassium hydrogen phosphate, 21.75 g dipotassium hydrogen phosphate, 33.4 g disodium hydrogen phosphate heptahydrate, and 1.7 g ammonium chloride in 500 ml distilled water. Dilute to one liter with distilled water.
 - 6.2.8 Potassium iodide solution: Dissolve 10 g reagent grade potassium iodide in 100 ml distilled water. Prepare this solution only when needed.
 - 6.2.9 Prepare concentration according to the level of chlorine in sample. Volume added should be less than one percent of sample volume.
 - 6.2.10 Sulfuric acid solution (1 N): Dilute 29 ml reagent grade concentrated sulfuric acid in one liter distilled water.
- 6.3 Glucose--glutamic acid solution: Dry reagent grade glucose and reagent grade glutamic acid at 103° C. for one hour. Add 150 mg glucose and 150 mg glutamic acid to distilled water and dilute to one liter. Sterilize in autoclave and dispense into 100 ml storage bottles. Store in 4° C. refrigerator.

7. Procedure

7.1 Standardization of the Dissolved Oxygen Meter and Probe

- 7.1.1 Mix the distilled water stored for preparing BOD dilution to ensure a uniform concentration of dissolved oxygen.
- 7.1.2 Discard the first 300 ml of the distilled water drawn through the tygon tubing that is attached to the distilled water carboy. Using the tygon tubing, fill three 275 ml BOD bottles with a minimum of surface agitation and entrained air. Fill each bottle to overflowing and cap immediately.
- 7.1.3 To two of the bottles, deliver 2 ml manganese sulfate solution and then 2 ml alkali-iodide-azide reagent below the surface using a serological pipet. Cap immediately and invert the bottles at least 15 times. When the precipitation has settled (2/3 of bottle contains clear supernatant), invert again at least 15 times. Allow the precipitate to settle, then add 2 ml of concentrated sulfuric acid, stopper immediately, and invert until the floc completely dissolves. The solution should be a clear, yellowish-brown in color.
- 7.1.4 Place the entire solution into a 500 ml Erlenmeyer flask and titrate with standardized sodium thiosulfate solution to a pale straw color. Add one ml starch solution and complete the titration until the blue color just disappears. Titrate the solution in the second BOD bottle. Use the average of the two titrations for the dissolved oxygen concentration of the distilled water.
- 7.1.5 Check the dissolved oxygen meter according to the manufacturer's instructions for battery charge and instrument zero. Check the probe membrane for tears, wrinkles, or bubbles. Replace the membrane and filler solution if these conditions occur, or at least every two months.
- 7.1.6 Establish the true zero for the meter-probe combination by placing the probe in a BOD bottle containing distilled water and an excess of reagent grade sodium sulfite. Rinse the probe with distilled water after this step has been completed.
- 7.1.7 Calibrate the meter-probe combination using the third BOD bottle containing distilled water that was collected in step 7.1.2 above. This

calibration procedure should be performed each day the meter-probe combination is used. Calibration is good for approximately four hours.

- 7.1.8 Store the probe in a BOD bottle filled with distilled water.

7.2 Sample Pre-treatment.

- 7.2.1 Temperature: Warm samples to room temperature before proceeding with the analysis.

- 7.2.2 pH: If the pH of the sample is not between 6 and 8, then it must be neutralized before BOD dilutions are made. The pH adjustment is made with 1 N sulfuric acid or 1 N sodium hydroxide to a pH 7.

- 7.2.3 Chlorinated samples: Samples should not contain residual chlorine. The following procedure should be used to detect and remove the residual chlorine before BOD analysis:

- a. To a 100 ml aliquot of well mixed sample, add sufficient 2 percent H_2SO_4 to adjust pH to 4, add one scoop of potassium iodide crystals, and one ml starch solution. If a blue color develops, titrate with sodium sulfite solution until the blue color just disappears.
- b. To a measured quantity of well mixed sample which is sufficient to prepare BOD dilutions, add sodium sulfite solution in the proportions determined in 7.2.3 a. Shake the sample to remove the residual chlorine and to help oxidize any excess sodium sulfite.

- 7.2.4 Supersaturation: Any water sample with a dissolved oxygen of 9 mg/l or more is considered supersaturated and it must be corrected before the BOD dilutions are made. Transfer a quantity of sample, which will be used for BOD to a clean, dry bottle. Shake the sample vigorously until the excess dissolved oxygen is removed.

- 7.2.5 Seed: The sample dilutions of chlorinated samples, strongly acidic or basic samples, and many industrial wastes may not contain a sufficient number of microorganisms to produce reliable results and must be seeded with organisms by the addition of a known quantity of settled sewage to the sample dilution.

- a. As a general rule, seed all sample dilutions which have been chlorinated, neutralized, or collected from industrial wastes.
- b. Use seed from settled domestic wastewater that has been stored at 20° C. for 24-36 hours.

7.3 Preparation of Sample Dilutions

- 7.3.1 Dilution water: To a carboy of distilled water which has been stored at 20° C. (6.2.4), add one ml/l of each: phosphate buffer solution (6.2.7), magnesium sulfate solution (6.2.6), calcium chloride solution (6.2.3), and ferric chloride solution (6.2.5). Mix and discard the first 300 ml of this solution that is dispensed through the attached tygon tubing.
- 7.3.2 The number and extent of sample dilutions taken depends on the expected strength of the sample. As a rough guide, the following ranges of BOD₅ values can be expected for the types of samples shown:

<u>Sample Type</u>	<u>Expected BOD (Range)</u>
Surface Water	0-20 mg/l
Polluted Surface Water	10-50
Sewage (Treated Effluent)	10-500
Sewage (Domestic)	100-500
Industrial Waste	10-500
Strong Industrial Waste	500-5,000
Slaughterhouse, Dairy, and Feedlot Wastes (Untreated)	1,000-20,000

The sample should be diluted so that a residual D.O. of at least one mg/l remains after five days incubation and the uptake of dissolved oxygen at least 2 mg/l occurs. Several dilutions of the sample are prepared to obtain dissolved oxygen uptake in this range.

- 7.3.3 Dilutions greater than 1:100: Make a primary dilution of the sample in a graduated cylinder and the final dilution directly in the bottle.

Dilutions less than 1:100: Place the volume of sample directly into the bottle and if needed, add one ml of seed (7.2.5) to the BOD bottle. Slowly fill the remainder of the bottle with dilution water (7.3.1) so that the insertion of the stopper displaces any possible air, leaving no bubbles.

7.4 Seed

- 7.4.1 It is necessary to have present a population of microorganisms capable of oxidizing the biodegradable organic matter in the sample. Each sample which might be deficient in microbial population (7.2.5) must have additional seed material. This is done by placing one ml of seed material directly into the bottle before the dilution water is added and this seed should contribute between 0.6 and one mg/l in the oxygen uptake if the BOD of the seed is approximately 200 mg/l.
- 7.4.2 If the samples are seeded, a BOD must be run on the seed material and the five-day oxygen uptake must be used to correct the seeded sample dilutions. The seed material is diluted to a proportion which will produce a residual D.O. of at least one mg/l and a D.O. depletion of at least 2 mg/l. An initial D.O. is obtained at the same time the sample dilutions are read, the seed dilution is incubated for five days, and the final D.O. is read at the same time as the samples.

7.5 Dissolved Oxygen (D.O.) Readings and Sample Incubation

- 7.5.1 The initial dissolved oxygen is read on each sample dilution by the membrane electrode method. Any sample volume which has been lost in reading the dissolved oxygen should be replaced with dilution water.
- 7.5.2 The sample is stoppered tightly and incubated for five days at 20° C. The water seal which is required during incubation is obtained by inverting the BOD bottle in a pan which contains water.
- 7.5.3 After a five-day incubation period, the final dissolved oxygen reading is obtained for all sample dilutions.

7.6 Dilution Water Blank

Use dilution water blanks as a rough check on the quality of the unseeded dilution water and the cleanliness of the incubation bottles. When making initial sample dilutions, the first and last BOD bottle should be used as dilution water blanks. Intermittant blanks should be used if the number of samples is large or there is a change in the dilution water bottles. Initial D.O. readings should be taken at the same time as the sample dilutions are read

(7.5.1) and final D.O. readings are taken after the five-day incubation (7.5.3). The D.O. uptake should not be greater than 0.2 mg/l or there is a problem in the quality of the dilution water.

7.7 Glucose--Glutamic Acid Check

7.7.1 A mixture of glucose-glutamic acid is analyzed for BOD with each sample run. The measurement of the pure organic compounds will give an indication of the dilution water quality, seed effectiveness, and the analytical technique.

7.7.2 A 1.45 percent and 2.54 percent solution of the glucose-glutamic acid solution (6.3) is made, seed added, and the five-day BOD is obtained as outlined in 7.5. If the five-day BOD value of the check is outside 200 ± 37 mg/l, reject any BOD determinations made with the seed and dilution water and seek the cause of the problem.

8. Calculations

8.1 The sample description, lab number, date and values for D.O. readings, and dilutions are recorded on the bench sheet.

8.2 Calculations of the five-day BOD for samples not seeded, the seed dilution, and the dilution water blank:

$$\text{BOD}_5 = (D_1 - D_2)/P$$

D_1 = initial D.O. reading

D_2 = final D.O. reading after five-day incubation

P = decimal fraction of the sample used to make the sample dilution.

8.3 Calculation of five-day BOD for the glucose-glutamic acid dilution and the seeded sample:

$$\text{BOD}_5 = [(D_1 - D_2) - (B_2 - B_1) F]/P$$

P and $D_1 - D_2$ defined in 8.2

$B_2 - B_1$ = depletion of D.O. of the seed for five days

$$F = \frac{\% \text{ seed in sample dilution}}{\% \text{ seed in seed dilution}}$$

9. Quality Control

9.1 Internal Quality Control

- 9.1.1 Glucose-glutamic acid solutions (1.45 percent and 2.54 percent) are analyzed for five-day BOD. The results are collected, treated statistically, and control limits are determined. The control limits are evaluated with each analytical run.
- 9.1.2 The blank dilution water is analyzed for the five-day period. This shows the presence of organic contamination in the system. These data are collected and treated statistically to provide control information.
- 9.1.3 Duplicate field samples and duplicate lab samples are analyzed and the data is collected for statistical evaluation. Control limits are placed on the analyses to provide adequate precision in the test.

9.2 External Quality Control

- 9.2.1 Regular participation in annual interlaboratory audits are sponsored by USEPA, Region V. These are audits on the performance of the five-day BOD procedure.

10. References

- 10.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1985, p. 525, Method 507.
- 10.2 Chemical Analyses for Water Quality, Training Course Manual, U.S. Department of the Interior, Federal Water Pollution Control Administration, pp. 6-1 to pp. 8-13.
- 10.3 Methods for Chemical Analysis of Water and Wastes, EPA, 1983, Method 405.1.

M-6
Chloride

Modifications per U.S. EPA

6.5 Add one more calibration standard as follows:

<u>ml of Stock Chloride Solution</u>	<u>Conc. mg/Cl/l</u>
5	5

CHLORIDE
(Automated Ferricyanide Method)
(16th Ed. Std. Methods-ISBN Modifications)

ISBN Code No. C1-C-1-88
STORET No. 00940
Approved for NPDES

1. Scope of Application

- 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. The applicable range is 5-100 mg/l Cl. Approximately 40 samples per hour can be analyzed.

2. Summary of Method

- 2.1 Thiocyanate ion (SCN) is liberated from mercuric thiocyanate, through sequestration of mercury by chloride ion to form unionized mercuric chloride. In the presence of ferric ion, the liberated SCN forms highly colored ferric thiocyanate, in concentration proportional to the original chloride concentration.

3. Sample Handling and Preservation

- 3.1 The samples are collected in one liter polyethylene bottles. No preservative is needed.
- 3.2 Holding time is 28 days.

4. Comments

- 4.1 No significant interferences.

5. Apparatus

- 5.1 No significant change from referenced method.

6. Reagents

- 6.1 Stock mercuric thiocyanate solution: Place 500 ml of methanol in a one liter volumetric flask. Add 4.17 g of mercuric thiocyanate, $\text{Hg}(\text{SCN})_2$, and dissolve. Dilute to volume with methanol, mix, and filter through filter paper.
- 6.2 Stock ferric nitrate solution: Place 202 g of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in a 1 liter volumetric flask and add approximately 500 ml of distilled water. After dissolution, carefully add 22.2 ml of concentrated nitric acid to the flask and mix. Dilute to volume, mix, and filter through filter paper. Store in an amber reagent bottle.

- 6.3 Color reagent (prepare fresh daily): Place 75 ml of mercuric thiocyanate stock solution into a 500 ml volumetric flask. Add 75 ml of the stock ferric nitrate solution, dilute to volume with distilled water, and mix well.
- 6.4 Stock chloride solution: Place 0.8241 g NaCl dried at 140° C. in distilled water and dilute to 1 liter;
1 ml = 0.5 mg Cl.
- 6.5 Prepare a series of working standards by diluting suitable volumes of stock chloride solution to 500 ml with distilled water. The following dilutions are suggested:

<u>ml of stock chloride solution</u>	<u>conc. mg Cl/l</u>
5	5
10	10
20	20
40	40
60	60
80	80
100	100

- 6.6 Dilution water: Add Brij-35 to distilled water (5 drops per liter).

7. Procedure

- 7.1 No advance sample preparation is required. The manifold is set up as shown in Figure 1.
- 7.2 After the colorimeter and recorder warm up for approximately 30 minutes, establish a reagent baseline.
- 7.3 Place working standards in sampler in order of increasing concentrations. Complete filling of sampler tray with samples to be analyzed.

8. Calculations

- 8.1 The AutoAnalyzers are connected to a computer which receives the response signal from the colorimeter. After the type of curve fit is selected by the operator, the computer calculates the calibration curve by least squares method and generates concentration values for the samples, quality control solution, and laboratory blanks.
- 8.2 The response signal from the colorimeter is also connected to a strip chart recorder. The chart can be used to calculate concentration values by use of the overlay. The standard curve is prepared on the overlay by plotting the peak heights of standards against known concentrations. The concentration of the samples are obtained by comparing sample peak heights with the standard curve. The standard curve is not linear throughout the working range.

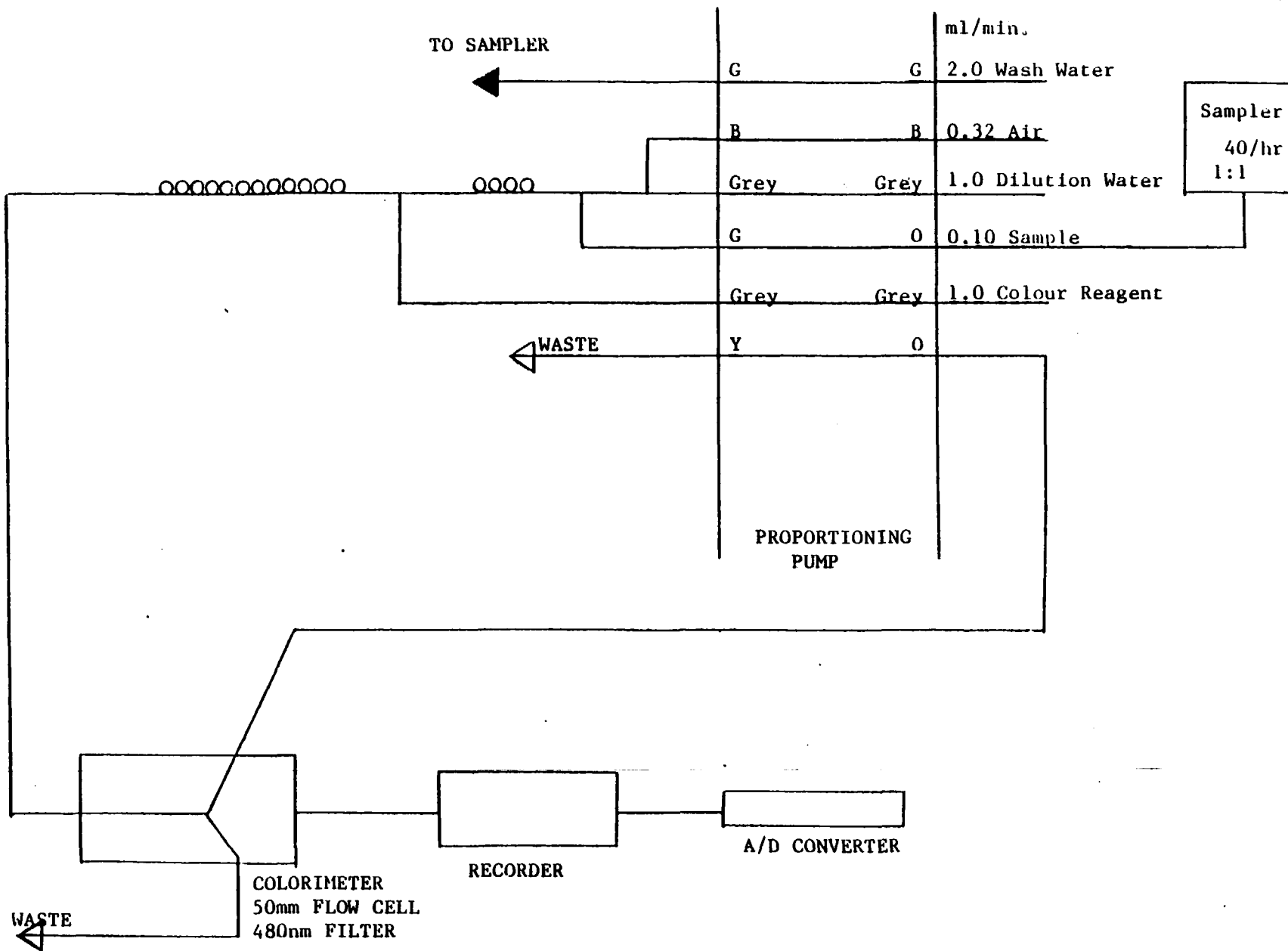
9. Quality Control

- 9.1 The following are analyzed every 20 samples:
1. Quality control sample.
 2. Spiked sample.
 3. Duplicate.
- 9.2 The laboratory blank, field blank and field duplicates are analyzed on a routine basis.
- 9.3 Internal audits using USEPA ampules.
- 9.4 Interlaboratory studies sponsored by the USEPA.

10. References

- 10.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 292, Method 407D, 1985.
- 10.2 Federal Register, Vol. 41, No. 232-Wednesday, December 1, 1976, p. 52781
- 10.3 Methods for Chemical Analysis of Water and Wastes, 1983, p. 325.2, USEPA
- 10.4 J. E. O'Brian, Automatic Analysis of Chlorides in Sewage, Waste Eng., 33, 670-672 (Dec. 1962)

ENV LABS 3
2-4-88
NAC
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CHLORIDE MANIFOLD
(FERRICYANIDE METHOD)
AUTOANALYZER II

(MODIFIED 11-19-85)

CHEMICAL OXYGEN DEMAND (Low Level)
(EPA Method, 1971)

ISBH Code No. COD-B-10-88
STORET No. 00335
EPA Approved

1. Scope and Application

- 1.1 The scope of this modification of the Chemical Oxygen Demand test is the same as for the high level test. It is applicable to the analysis of surface waters, domestic and industrial wastes with low demand characteristics.
- 1.2 This method (low level) is applicable for samples having a COD in the range of 5-80 mg/l COD.

2. Summary of Method

- 2.1 Organic and oxidizable inorganic substances in an aqueous sample are oxidized by potassium dichromate solution in 50 percent (by volume) sulfuric acid solution. The excess dichromate is titrated with standard ferrous ammonium sulfate using orthophenanthroline ferrous complex (ferroin) as an indicator.

3. Sampling and Preservation

- 3.1 Collect the samples in glass bottles, if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.
- 3.2 Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.
- 3.3 Samples may be preserved with sulfuric acid at a rate of 2 ml of 50 percent H_2SO_4 per liter of sample.

4. Interferences

- 4.1 Traces of organic material either from the glassware or atmosphere may cause a gross, positive error.
 - 4.1.1 Extreme care should be exercised to avoid inclusion of organic materials in the distilled water used for reagent preparation or sample dilution.

4.1.2 Glassware used in the test should be conditioned by running blank procedures to eliminate traces of organic material.

4.2 Volatile materials may be lost when the sample temperature rises during the sulfuric acid addition step.

4.3 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to the digestion flask to complex the chlorides, thereby effectively eliminating the interference on all but brine and estuarine samples.

5. Apparatus

5.1 Reflux apparatus--Glassware should consist of a 250 ml erlenmeyer flask or a 300 ml round bottom flask made of heat-resistant glass connected to a 12-inch Allihn condenser by means of a ground-glass joint. Any equivalent reflux apparatus may be substituted provided that a ground-glass connection is used between the flask and the condenser.

6. Reagents

6.1 Distilled water. Special precautions should be taken to insure that distilled water used in this test be low in organic matter.

6.2 Standard potassium dichromate solution (0.025N)--Dissolve 12.259 g $K_2Cr_2O_7$, primary standard grade, previously dried at $103^\circ C$. for two hours, in distilled water and dilute to 1.0 liter. Mix this solution thoroughly, then dilute 100 ml to 1.0 liter with distilled water.

6.3 Sulfuric acid reagent--Conc. H_2SO_4 containing 22 g silver sulfate, Ag_2SO_4 , per nine-pound bottle (one to two days required for dissolution).

6.4 Standard ferrous ammonium sulfate (0.01N)--Dissolve 39 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water. Add 20 ml of conc. H_2SO_4 , cool, and dilute to 1.0 liter. Dilute 100 ml of this solution to 1.0 liter with distilled water. This solution must be standardized daily against 0.025N $K_2Cr_2O_7$ solution.

6.4.1 Standardization--Dilute 10.0 ml standard $K_2Cr_2O_7$ solution to about 100 ml. Add 30 ml of H_2SO_4 and cool. Titrate with ferrous ammonium sulfate using two to three drops of ferroin indicator. The color change is sharp, going from blue-green to reddish-brown.

$$\text{Normality} = \frac{(\text{ml } K_2Cr_2O_7)(0.025)}{\text{ml } Fe(NH_4)_2(SO_4)_2}$$

- 6.5 Mercuric sulfate--Powdered $HgSO_4$.
- 6.6 Phenanthroline ferrous sulfate (ferroin) indicator solution--Dissolve 1.48 g of 1-10-(ortho)-phenanthroline monohydrate, together with 0.70 g of $FeSO_4 \cdot 7H_2O$ in 100 ml of water. This indicator may be purchased already prepared.
- 6.7 Silver sulfate--Powdered Ag_2SO_4 .
- 6.8 Sulfuric acid (sp. gr. 1.84)--Concentrated H_2SO_4 .

7. Procedure

- 7.1 Place 1 g $HgSO_4$ in the reflux flask.
- 7.2 Add 20 ml of sample into the 250 ml erlenmeyer flask and swirl to mix.
- 7.3 Add 10 ml of 0.025N $K_2Cr_2O_7$ and swirl to mix.
- 7.4 Slowly add 30 ml of conc. H_2SO_4 - $AgSO_4$ down the inside wall of the reflux flask.
- 7.5 Attach the flask to the condenser, swirl to mix, and reflux for two hours.
- 7.6 After refluxing, allow the flask to cool and wash down the condenser with about 25 ml of distilled water. Dilute the acid solution to about 140 ml with distilled water and allow the solution to cool to room temperature.
- 7.7 Add two to four drops of ferroin indicator to the solution and titrate the excess dichromate with 0.01N ferrous ammonium sulfate.
- 7.8 Simultaneously run a blank determination using 20 ml of distilled water in place of sample.

8. Quality Control

- 8.1 The following are analyzed for every 20 samples:
 - 1. Quality control samples.
 - 2. Duplicates.
 - 3. Spiked samples.
 - 4. Blanks.

8.2 Internal audits using USEPA ampules.

8.3 Interlaboratory studies sponsored by USEPA.

9. References

- 9.1 "Standard Methods for the Examination of Water and Wastewater," 14th Edition, page 550, Method 508.
- 9.2 "Methods for Chemical Analysis of Water and Wastes," 1979, Method 410.
- 9.3 Federal Register, Vol. 38, No. 199 (October 16, 1973), Part II, EPA, Water Programs.

CHEMICAL OXYGEN DEMAND (High Level)
(EPA Method, 1971)

ISBH Code No. COD-A-10-88
STORET No. 00340
EPA Approved

1. Scope and Application

- 1.1 This method determines the quantity of oxygen required to oxidize the organic matter in a waste sample, under specific conditions of oxidizing agent, temperature, and time.
- 1.2 Since the test utilizes a rigorous chemical oxidation rather than a biological process, the result has no definable relationship to the BOD of the waste. The test results should be considered as an independent measurement of organic matter in the sample, rather than as a substitute for the BOD test.
- 1.3 The method can be applied to domestic and industrial waste samples having an organic carbon concentration greater than 15 mg/l. For lower concentrations of carbon such as in surface water samples, the Low Level Method should be used.

2. Summary of Method

- 2.1 Organic substances in the sample are oxidized by potassium dichromate in 50 percent sulfuric acid solution at reflux temperature. Silver sulfate is used as a catalyst and mercuric sulfate is added to remove chloride interference. The excess dichromate is titrated with standard ferrous ammonium sulfate, using orthophenanthroline ferrous complex as an indicator.

3. Sample Handling and Preservative

- 3.1 The analysis is performed as soon as possible.
- 3.2 The sample is preserved with 2 ml of 50 percent sulfuric acid per liter and refrigerated at 4° C.

4. Procedure

- 4.1 Pipet 20 ml of sample into a 250 ml erlenmeyer flask.
- 4.2 Add 0.14 g HgSO_4 .
- 4.3 Add 10 ml of 0.25N $\text{K}_2\text{Cr}_2\text{O}_7$ and swirl to mix.

- 4.4 Slowly add 30 ml of concentrated H_2SO_4 reagent (22 g Ag_2SO_4 per nine-pound bottle of acid) and swirl to mix. To reduce loss of volatile organics, the flask should be cooled during addition of the sulfuric acid solution.
- 4.5 Attach flask to condenser and reflux for two hours.
- 4.6 After refluxing, allow the flask to cool and wash down the condenser with about 25 ml of distilled water.
- 4.7 Dilute the acid solution in the flask to 140 ml with distilled water and allow the solution to cool to room temperature.
- 4.8 Add four drops of ferroin indicator to the solution and titrate the excess dichromate with 0.1N ferrous ammonium sulfate.
- 4.9 Simultaneously run a blank determination using 20 ml distilled water in place of sample.

5. Calculations

$$\text{COD, mg/l} = \frac{(A - B) N \times 8000}{S}$$

A = ml of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ solution required for titration of blank.

B = ml of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ solution required for titration of sample.

N = normality of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ solution.

S = ml of sample used for test.

6. Quality Control

6.1 The following are analyzed for every 20 samples:

1. Quality control samples.
2. Duplicates.
3. Spiked samples.
4. Blanks.

6.2 Internal audits using USEPA ampules.

6.3 Interlaboratory studies sponsored by USEPA.

7. References

- 7.1 Federal Register, Vol. 38, No. 199 (October 16, 1973),
Part II, EPA, Water Programs.
- 7.2 "Standard Methods for the Examination of Water and
Wastewater," 13th Edition, page 495, Method 220.
- 7.3 "Methods for the Chemical Analysis of Water and Wastes,"
1971, Environmental Protection Agency, page 17.

CHROMIUM, HEXAVALENT
(Standard Methods, 16th Edition)

ISBH Code No. H.Cr-A-1-88
STORET No. 01032
Approved for NPDES

1. Scope and Application

- 1.1 This method is applicable to the determination of chromium in surface waters and domestic and industrial wastes.
- 1.2 The method is applicable in the range 0.01 to 1.0 mg/l Cr.

2. Summary of the Method

- 2.1 Hexavalent chromium reacts with diphenylcarbazide in acidic medium to produce a red violet coloration of unknown composition suitable for determination of low concentrations of chromium.

3. Sample Handling and Preservation

- 3.1 Samples are collected in 2 liter plastic bottles and are not preserved.
- 3.2 Holding time is 24 hours.

4. Interferences

- 4.1 The interferences are the same as listed in the method for total chromium.

5. Apparatus

- 5.1 Spectrophotometer for use at 540 nm providing a light path of 1 cm or longer.
- 5.2 Filter, glass fiber.

6. Reagents

- 6.1 Chromium standards--Same as those listed in method for total chromium.
- 6.2 Sulfuric acid, 1 + 1.
- 6.3 Phosphoric acid, 85 percent.
- 6.4 Diphenylcarbazide solution--Dissolve 0.25 g 1,5-diphenylcarbazide in 50 ml acetone. Prepare fresh daily.

7. Procedure

- 7.1 Prepare the following standards in 50 ml volumes and transfer to 125 ml erlenmeyer flasks: 0, 0.05, 0.1, 0.3, 0.6 mg/l Hex. Cr.
- 7.2 Pipet two 50 ml aliquots (1 sample blank and 1 sample for the color reaction) of each sample into 125 ml Erlenmeyer flasks or 50 ml nessler tubes.
- 7.3 Add 0.5 ml 1 + 1 H_2SO_4 , 0.125 ml H_3PO_4 , and mix the samples and standards.
- 7.4 Add 1.0 ml of diphenylcarbazide solution to only one flask or tube of each sample set, mix, and allow to stand 5 minutes.
- 7.5 Measure the absorbance of the standards and samples at 540 nm in 1-inch cells.

8. Calculation

- 8.1 Prepare a standard curve from the absorbance readings of the standards.
- 8.2 Subtract the absorbance of the sample blank from the absorbance of the colorimetric product to give the turbidity correction. Read the concentration of the samples from the standard curve.

9. Comments

- 9.1 The sample can be filtered through glass fiber filter to remove excess turbidity or color. If the filtration does not remove this interference, a sample blank can be run by deleting the addition of diphenylcarbazide in the sample.
- 9.2 The procedure can be carried out in 50 ml nessler tubes in place of the 125 ml Erlenmeyer flasks.

10. Quality Control

- 10.1 The following are analyzed every twenty (20) samples:

1. Quality control samples.
2. Spiked samples.
3. Duplicates.
4. Blanks.

11. Reference

- 11.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 201, Method 312B, 1985.

ENVLABS 3
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CHROMIUM, TOTAL
(Standard Methods, 16th Edition)

ISBH Code No. T. Cr-B-2-88
STORET No. 01034
Approved for NPDES and SDWA

1. Scope and Application

- 1.1 This method is applicable to the determination of chromium in surface waters, domestic and industrial wastes, and saline waters.
- 1.2 The method is applicable in the range 0.01 to 1.0 mg/l Cr.

2. Summary of Method

- 2.1 The sample is digested with $\text{HNO}_3\text{-H}_2\text{SO}_4$ to decompose organic matter. The acidity of the sample is adjusted to 0.5 N and the chromium is oxidized to the hexavalent state with potassium permanganate. Addition of an excess of diphenylcarbazide yields a red-violet product, its absorbance at 540 mm is measured photometrically.

3. Sample Handling and Preservation

- 3.1 Samples are collected in 2 liter plastic bottles and are preserved with 10 ml conc. HNO_3 per 2 liter.

4. Interferences

- 4.1 Molybdenum and mercury interfere with color formation when in excess of 200 mg/l.
- 4.2 Vanadium interferes, but can be present in a concentration up to ten times that of chromium without causing trouble.
- 4.3 Ferric iron may interfere, but in the absence of chlorides and with sulfuric and phosphoric acid present, the ferric iron color is not strong and no difficulty is encountered if the absorbance is measured at the appropriate wavelength.
- 4.4 Large amounts of some metals cause low results by consuming the diphenylcarbazide reagent.
- 4.5 Interfering amounts of molybdenum, vanadium, iron, and copper can be removed by extraction of the cupferrates of these metals into chloroform.
- 4.6 High levels of chlorides can result in loss of chromium as chromyl chloride.

5. Apparatus

- 5.1 Spectrophotometer for use at 540 mμ providing a light path of one cm or longer.
- 5.2 Erlenmeyer flasks, 250 ml.
- 5.3 Filtering apparatus; flasks, filters, etc.

6. Reagents

- 6.1 Stock Chromium Solution: Dissolve 141.4 mg $K_2Cr_2O_7$ in distilled water and dilute to 1.0 liter; $1.00\text{ ml} = 50\text{ ug Cr}$.
- 6.2 Standard Chromium Solution: Dilute 10.0 ml stock chromium solution (6.1) to 100 ml; $1.00\text{ ml} = 5.0\text{ ug Cr}$.
- 6.3 Nitric Acid, conc.
- 6.4 Sulfuric Acid, 1 + 1.
- 6.5 Methyl Orange Indicator Solution.
- 6.6 Hydrogen Peroxide, 30 percent.
- 6.7 Ammonium Hydroxide, conc.
- 6.8 Phosphoric Acid, 85 percent.
- 6.9 Potassium Permanganate Solution: Dissolve 4.0 g $KMnO_4$ in 100 ml distilled water.
- 6.10 Sodium Azide Solution: Dissolve 0.5 g NaN_3 in 100 ml distilled water.
- 6.11 Diphenylcarbazide Solution: Dissolve 0.25 g 1,5-diphenylcarbazide in 50 ml acetone. Prepare fresh at time of analysis.

7. Procedure

- 7.1 Prepare working standards of 50 ml volumes (0, 0.05, 0.1, 0.3, 0.6 mg/l Cr) in 250 ml Erlenmeyer flasks.
- 7.2 Pipet 50 ml sample into 250 ml Erlenmeyer flasks.
- 7.3 Add one ml hydrogen peroxide, 2.5 ml conc. HNO_3 , 5 ml 1 + 1 H_2SO_4 .
- 7.4 Evaporate on a hot plate (in a hood) until dense white fumes of SO_3 appear in the flask, but do not continue heating beyond this point. If solution is not clear, add another 5 ml of HNO_3 and repeat evaporation to SO_3 fumes. Continue nitric acid treatment until the solution is clear or free of organic material.

- 7.5 Cool solution to room temperature and carefully add approximately 25 ml of distilled water.
- 7.6 Using methyl orange as an indicator, add conc. NH_4OH until the solution is just basic. Then add 1 + 1 H_2SO_4 dropwise until solution is acidic, plus one ml in excess.
- 7.7 Add 0.25 ml of H_3PO_4 and mix sample.
- 7.8 Heat solution to boiling and add two drops of potassium permanganate solution to give a dark red color. If fading occurs, add additional drops of KMnO_4 to maintain an excess of two drops.
- 7.9 Boil two minutes longer, add one ml of sodium azide solution, and continue gentle boiling. If red color does not fade completely in 30 seconds, add another one ml of sodium azide solution. Continue boiling for one minute after the color has faded completely.
- 7.10 Filter the sample through a glass fiber filter and bring the volume of the cooled solution to 50 ml (if sediment present).
- 7.11 Add one ml of diphenylcarbazide solution to the standards and samples, mix, and allow to stand 5-10 minutes for full color development.
- 7.12 Read the absorbance of the samples and standards on the spectrophotometer at 540 mμ in one cm cells.
8. Calculations
- 8.1 A standard curve is prepared and the concentration of chromium in the samples is read from the standard curve.
9. Comments
- 9.1 The samples which show color or turbidity after digestion and filtration must be run against a sample blank (absorbance reading taken before addition of the diphenylcarbazide).
10. Quality Control
- 10.1 The following are analyzed every 20 samples:
1. Quality control sample.
 2. Spiked sample.
 3. Duplicate.
- 10.2 Interlaboratory studies are also sponsored by the USEPA and used for external audits.

11. References

- 11.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 201, Method 312B (1985).
- 11.2 Federal Register, Vol. 38, No. 199, (October 16, 1973), Part II, EPA, Water Programs.
- 11.3 ASTM Standards, Part 23, 1973, p. 293, Method D 1687.

ENV LABS 4
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NITROGEN, AMMONIA
Colorimetric, Automated Phenate
(ISBH Modifications to EPA Method, 1979)

ISBH Code No. NH3-A-10-88
STORET NO. Total 00610
Approved for NPDES

1. Scope and Application
 - 1.1 This method covers the determination of ammonia in drinking, surface, and saline waters, domestic and industrial wastes in the range of 0.10 to 10 mg/l NH₃ as N. This range is for photometric measurements made at 630-660 nm in a 15 mm or 50 mm tubular flow cell. Higher concentrations can be determined by sample dilution. Approximately 20 to 60 samples per hour can be analyzed.
2. Summary of Methods
 - 2.1 Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside.
3. Sample Handling and Preservation
 - 3.1 Preservation by addition of 2 ml conc. H₂SO₄ per liter and refrigeration at 4 Deg. C.
4. Comments
 - 4.1 Calcium and magnesium ions may be present in concentration sufficient to cause precipitation problems during analysis. A sodium potassium tartrate solution is used to prevent the precipitation of calcium and magnesium ions from river water and industrial waste.
 - 4.2 Sample turbidity and color may interfere with this method. Turbidity must be removed by filtration prior to analysis. Sample color that absorbs in the photometric range used will also interfere.
5. Apparatus
 - 5.1 Technicon AutoAnalyzer Unit (AAII) consisting of:
 - 5.1.1 Sampler.
 - 5.1.2 Analytical Cartridge (AAII).
 - 5.1.3 Proportioning pump.
 - 5.1.4 Heating bath with double delay coil (AAI).
 - 5.1.5 Colorimeter equipped with 15 mm tubular flow cell and 630 nm filters.
 - 5.1.6 Recorder.
6. Reagents
 - 6.1 Distilled water: Special precaution must be taken to insure that distilled water is free of ammonia. Such water is prepared by passage of distilled water through an ion exchange column comprised of a mixture of both strongly acidic cation and strongly basic anion exchange resins. The regeneration of the ion exchange column should be carried out according to the instruction of the manufacturer.
NOTE 1: all solutions must be made using ammonia-free water.
 - 6.2 Sulfuric acid: 50% sulfuric acid.
 - 6.3 Sodium phenolate: Using a 1 liter Erlenmeyer flask, dissolve 30 ml phenol in 500 ml of distilled water. In small increments,

cautiously add with agitation, 40 ml of 50% NaOH. Periodically cool flask under water faucet. When cool, dilute to 1 liter with distilled water.

- 6.4 Sodium hypochlorite solution: Dilute 125 ml of a bleach solution containing 5.25% NaOCl to 250 ml with distilled water. Make fresh daily!
- 6.5 (Replace sodium potassium tartrate solution with the following EDTA reagent)
Disodium ethylenediamine-tetraacetate (EDTA) (5%): Dissolve 50g of EDTA (disodium salt) and 20 ml 50% sodium hydroxide in 1 liter of distilled water. Add 6 drops of Brij 35.
- 6.6 Sodium nitroprusside (0.05%): Dissolve 0.5 g of sodium nitroprusside in 1 liter of distilled water.
- 6.7 Stock solution: Dissolve 3.819 g of anhydrous ammonium chloride, NH_4Cl , dried at 105 Deg.C, in distilled water, and dilute to 1000 ml. (1.0 ml = 1.0 mg $\text{NH}_3\text{-N}$).
- 6.8 Standard Solution: Dilute 10.0 ml of stock solution (6.7) to 100 ml with distilled water. (1.0 ml = 0.10 mg $\text{NH}_3\text{-N}$).
- 6.9 Using standard solution, prepare the following standards:
- | $\text{NH}_3\text{-N}$, mg/l | ml Standard Solution/Vol D.W. |
|-------------------------------|-------------------------------|
|-------------------------------|-------------------------------|

0.1	1/1000 ml
0.5	1/200 ml
2.0	4/200 ml
5.0	10/200 ml
7.5	15/200 ml
10.0	20/200 ml

NOTE 2: When saline water samples are analyzed, Substitute Ocean Water (SOW) should be used for preparing the above standards used for the calibration curve; otherwise, distilled water is used. If SOW is used, subtract its blank background response from the standards before preparing the standard curve.

Substitute Ocean Water (SOW)

NaCl	24.53 g/l	NaHCO_3	0.20 g/l
MgCl_2	5.20 g/l	KBr	0.10 g/l
Na_2SO_4	4.09 g/l	H_3BO_3	0.03 g/l
CaCl_2	1.16 g/l	SrCl_2	0.03 g/l
KCl	0.70 g/l	NaF	0.003 g/l

- 6.10 The working standards for low level nitrate analysis are 2.0, 1.5, 1.0, 0.5, 0.1mg $\text{NH}_3\text{-N/l}$. The only modification of the manifold is disconnecting the dilution loop.
- 6.11 Wash water (dilution water): Add 2ml 50% sulfuric acid to 1 liter of distilled water and mix.

7. Procedure

- 7.1 Since the intensity of the color used to quantify the concentration is pH dependent, the acid concentration of the wash water and the standard ammonia solutions should approximate that of the samples. For example, the samples have been preserved with 2 ml 50% H₂SO₄/liter.
- 7.2 For a working range of 0.1 to 10. mg NH₃-N/l (AAII), set up the manifold as shown in figure 2. Higher concentrations may be accommodated by sample dilution.
- 7.3 Allow both colorimeter and recorder to warm up for 30 minutes. Obtain a stable baseline with all reagents, feeding distilled water through sample line.
- 7.4 For the AAII use a 40/hr 2:1 cam with a common wash.
- 7.5 Arrange ammonia standards in sampler in order of increasing concentration of nitrogen. Complete loading of sampler tray with unknown samples.

8. Calculations

- 8.1 The AutoAnalyzers are connected to a computer which receives the response signal from the colorimeter. After the type of curve fit is selected by the operator, the computer calculates the calibration curve by least squares method and generates concentration values for the samples, quality control solution, and laboratory blanks.
- 8.2 The response signal from the colorimeter is also connected to a strip chart recorder. The chart can be used to calculate concentration values by use of the overlay. The standard curve is prepared on the overlay by plotting the peak heights of standards against known concentrations. The concentrations of the samples are obtained by comparing sample peak heights with the standard curve. The standard curve is not linear throughout the working range.

9. Quality Control

- 9.1 The following are analyzed for every 20 samples:

1. Quality control samples
2. Duplicates
3. Spiked samples
4. Blanks

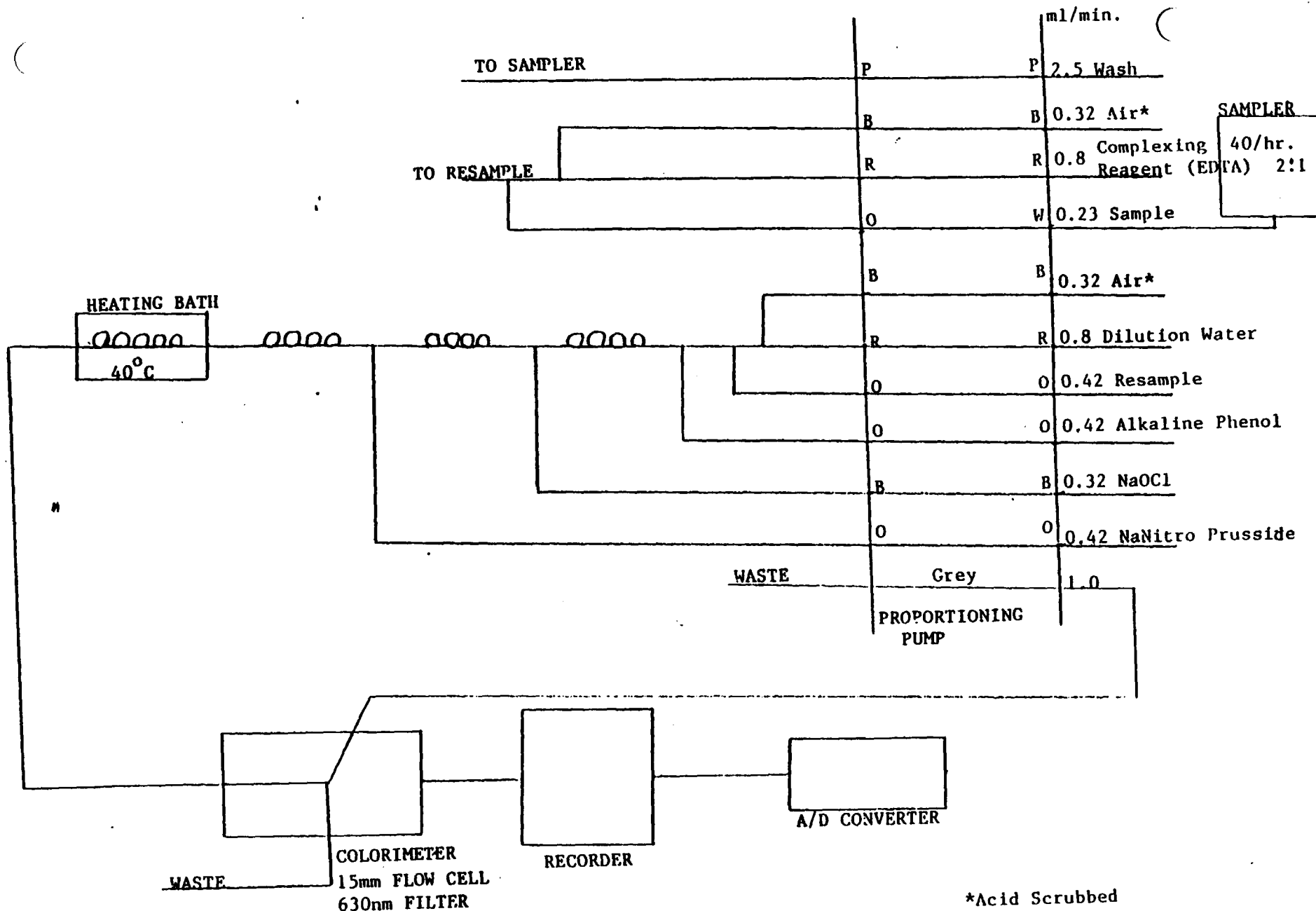
- 9.2 Internal audits using USEPA ampules.

- 9.3 Interlaboratory studies sponsored by USEPA.

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Nitrogen, Ammonia

Autoanalyzer II

(Modified 11-15-85)

FIGURE 2

NITROGEN, NITRATE + NITRITE
Colorimetric, Automated Cadmium Reduction
ISEH Modifications to EPA Method, 1979

ISEH Code No. NO3+NO2 (N)-E-10-88

STORET No. Total 00630

Approved for NPDES and SDWA

1. Scope and Application
 - 1.1 This method pertains to the determinations of nitrite singly, or nitrite and nitrate combined in surface and saline waters, and domestic and industrial wastes. The applicable range of this method is 0.1 to 10.0 mg/l nitrate+nitrite nitrogen. The range may be extended with sample dilution.
2. Summary of Method
 - 2.1 A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically. Separate, rather than combined nitrate+nitrite, values are readily obtained by carrying out the procedure first with, and then without the Cu-Cd reduction step.
3. Sample Handling and Preservation
 - 3.1 Analysis should be made as soon as possible. If analysis can be made within 24 hours, the sample should be preserved by refrigeration at 4 Deg. C. When samples must be stored for more than 24 hours, they should be preserved with sulfuric acid (2 ml 50% H2SO4 per liter) and refrigeration.
4. Interferences
 - 4.1 Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample may be pre-filtered.
 - 4.2 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.
5. Apparatus
 - 5.1 Technicon AutoAnalyzer (AAII) consisting of the following components:
 - 5.1.1 Sampler
 - 5.1.2 Analytical Cartridge (AAII)
 - 5.1.3 Proportioning Pump
 - 5.1.4 Colorimeter equipped with a 15 mm tubular flow cell and 520 nm filters.
 - 5.1.5 Recorder.
 - 5.1.6 A/D Converter and Computer.
6. Reagents
 - 6.1 Granulated cadmium: 20 mesh MCB Reagents.
 - 6.2 Cu - Cd column:
 - 6.2.1 The cadmium granules (new or used) are cleaned with 50% reagent grade HCl and then rinsed with distilled water. The color of the cadmium so treated should be silver.
 - 6.2.2 Swirl approximately 10g cadmium in 10 ml aliquots of 2% CuSO4.5 H2O for at least ten 30 sec. periods.

- 6.2.3 Wash the cadmium with distilled water (at least 10 times) to remove the precipitated copper. The color of the cadmium should be black.
- 6.3 Preparation of reduction column AIII: The reduction column is a U-shaped, 35 cm length, 2 mm I.D. glass tube (Note 1). Fill the reduction column with distilled water to prevent entrapment of air bubbles during the filling operations. Transfer the copper-cadmium granules (6.2) to the reduction column and place a glass wool plug in each end. To prevent entrapment of air bubbles in the reduction column be sure that all pump tubes are filled with reagents before putting the column into the analytical system.
- NOTE 1: A 0.081 I.D. pump tube (purple) can be used in place of the 2 mm glass tube.
- 6.4 Distilled water: Because of possible contamination, this should be prepared by passage through an ion exchange column comprised of a mixture of both strongly acidic-cation and strongly basic-anion exchange resins. The regeneration of the ion exchange column should be carried out according to the manufacturer's instructions.
- 6.5 Color reagent: To approximately 100 ml of distilled water, add, while stirring, 40g sulfanilamide, 2.0g N-1-Naphthyl-ethylenediamine-dihydrochloride, and 100ml concentrated phosphoric acid. Stir until dissolved and dilute to 1 liter.
- 6.6 Dilute hydrochloric acid, 1N: Dilute 8.3 ml of conc. HCl to 100 ml of distilled water.
- 6.7 Copper sulfate solution, 2%: Dissolve 20 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in 500 ml of distilled water and dilute to 1 liter. (Making the 2% CuSO_4 soln. slightly acidic by the addition of HCl improves the copper coating process.)
- 6.8 Wash solution: Use distilled water for unpreserved samples. For samples preserved with H_2SO_4 , use 2 ml 50% H_2SO_4 per liter of wash water. (Wash water is also used as dilution water.)
- 6.9 Ammonium chloride soln: Dissolve 85g of reagent grade ammonium chloride in 100ml of distilled water. Add 0.5 ml Brj-35 and dilute to 1 liter.
- 6.10 Stock nitrate solution: Dissolve 7.218 g KNO_3 and dilute to 1 liter in a volumetric flask with distilled water. Preserve with 2 ml of 50% H_2SO_4 per liter. Solution is stable for 6 months. (1 ml = 1.0 mg $\text{NO}_3\text{-N}$).
- 6.11 Stock nitrite solution: Dissolve 6.072 g KNO_2 in 500 ml of distilled water and dilute to 1 liter in a volumetric flask. Preserve with 2 ml of chloroform and keep under refrigeration. (1.0 ml = 1.0 mg $\text{NO}_2\text{-N}$).
- 6.12 Standard nitrate solution: Dilute 10ml of stock nitrate solution (6.10) to 100ml using distilled water. (1ml = 100ug $\text{NO}_3\text{-N}$).
- 6.13 Standard nitrite solution: Dilute 10.0 ml of stock nitrite (6.11) solution to 1000 ml (1.0 ml = 0.01 mg $\text{NO}_2\text{-N}$). Solution is unstable; prepare as required.
- 6.14 Working standards: Using the standard nitrate solution (6.12), prepare the following standards in volumetric flasks:

Conc. mg $\text{NO}_3\text{-N/l}$

ml std. soln/Vol DW

0.1

1 ml/l

0.5	1/200 ml
2.0	4/200 ml
5.0	10/200 ml
7.0	14/200 ml
10.0	20/200 ml

6.15 Sodium hydroxide solution, 0.5%: Add 10ml of 50% sodium hydroxide to 500ml distilled water and dilute to 1 liter. Make fresh daily!

7. Procedure

- 7.1 If the pH of the sample is below 5 or above 9, adjust to between 5 and 9 with either conc. HCl or conc. NH₄OH.
- 7.2 Set up the manifold as shown in Figure 1 (AAII). Care should be taken not to introduce air into reduction column on the AAII.
- 7.3 Allow both colorimeter and recorder to warm up for 30 minutes. Obtain a stable baseline with all reagents feeding distilled water through the sample line.
Note 3: Condition column by running 10 mg/l standard for 30 minutes if a new reduction column is being used. Subsequently wash the column with reagents for 20 minutes.
- 7.4 Place appropriate nitrate and/or nitrite standards in sampler in order of increasing concentration of nitrogen. Complete loading of sampler tray with unknown samples.
- 7.5 For the AAII, use a 40/hr., 2:1 cam.
- 7.6 Switch the sample line to sampler and start analysis.
- 7.7 After analysis, remove reduction column before cleaning the system.
- 7.8 For low level nitrate analysis, use standards of 2.0, 1.5, 1.0, 0.5, 0.1 mg/l. Disconnect the dilution loop and keep the remaining manifold unchanged.

8. Calculations

- 8.1 The AutoAnalyzers are connected to a computer which receives the response signal from the colorimeter. After the type of curve fit is selected by the operator, the computer calculates the calibration curve by least squares method and generates concentration values for the samples, quality control solution, and laboratory blanks.
- 8.2 The response signal from the colorimeter is also connected to a strip chart recorder. The chart can be used to calculate concentration values by use of the overlay. The standard curve is prepared on the overlay by plotting the peak heights of standards against known concentrations. The concentration of the samples are obtained by comparing sample peak heights with the standard curve. The standard curve is not linear throughout the working range.

9. Quality Control

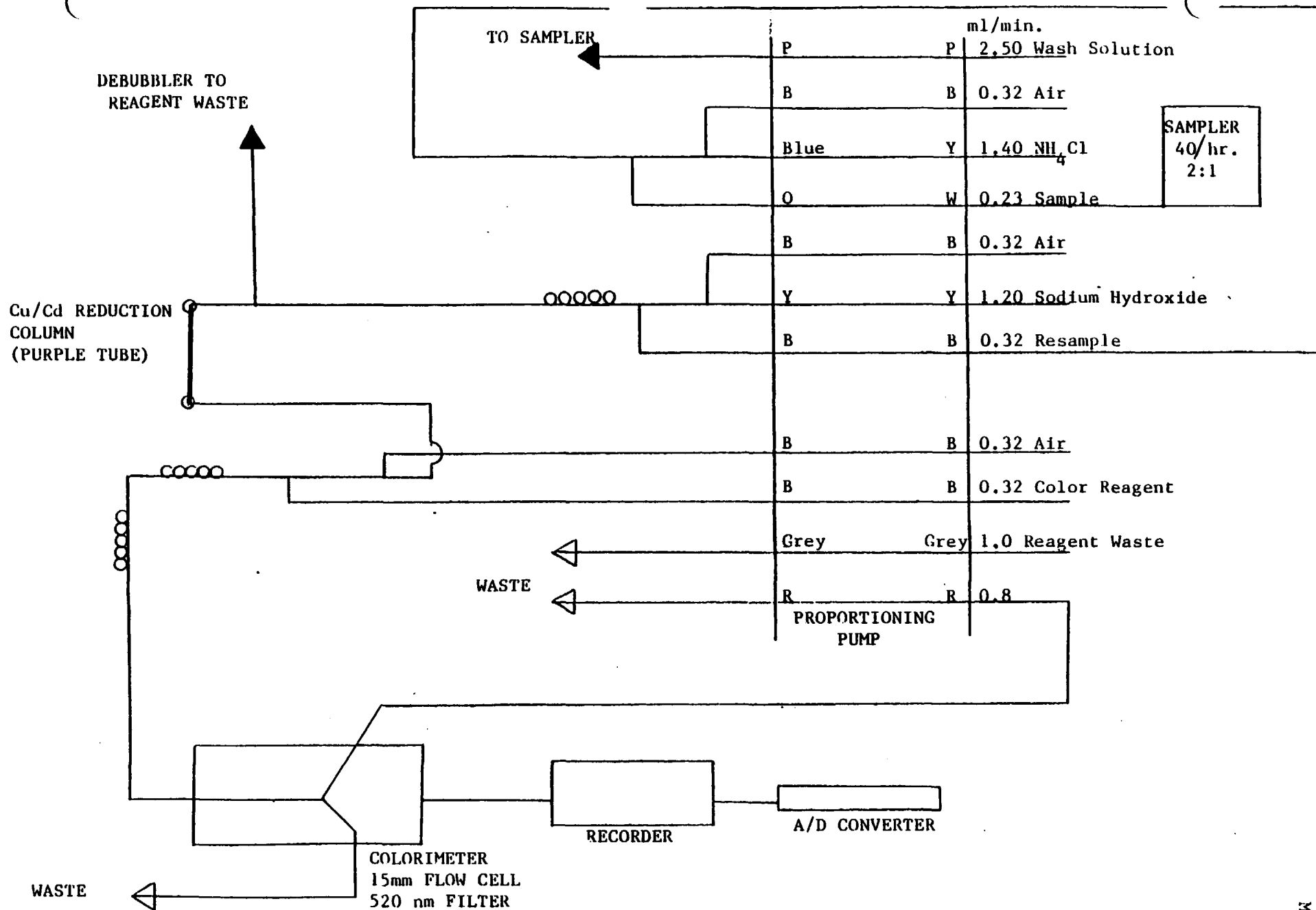
9.1 The following are analyzed for every 20 samples :

1. Quality control samples
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- 9.2 Internal audits using USEPA ampules.
- 9.3 Interlaboratory studies sponsored by USEPA.

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NITROGEN-NITRATE, NITRITE

AUTOANALYZER II

(MODIFIED 11-20-85)

FIGURE 1

NITROGEN, TOTAL KJELDAHL
(Ultramicro Semiautomated Method)

ISBH Code No. TKN-B-1-88
STORET NO. 00625

1. Scope and Application

- 1.1 This method is applicable to drinking water, surface water, domestic and industrial wastes.
- 1.2 The digested samples are analyzed by automated spectrophotometry at the rate of approximately 40 samples per hour.
- 1.3 The working range for the nitrogen is 0.1 to 10 mg/l, however, this range can be altered by modification of the digest volume or the manifold configuration.

2. Summary of Method

- 2.1 The manual digestion of the organic nitrogen is done in a Technicon block digester. The method of choice is the use of fuming sulfuric acid in the presence of mercuric oxide catalyst to convert the organic nitrogen compounds to ammonia. The addition of potassium sulfate to the Kjeldahl method increases the digestion rate. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds, of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semi-carbazones and some refractory tertiary amines.
- 2.2 The digested nitrogen compounds are analyzed for ammonia by a modification of the automated phenate method. In the phenate method, the indophenol blue reaction occurs as the ammonia reacts with the phenol and the hypochlorite to form a blue color. Sodium nitroprusside is used to intensify the color.

3. Sample Handling and Preservation

- 3.1 Samples should be preserved with 2 ml of 50 percent H_2SO_4 per liter. Preserved samples should be analyzed as soon as possible.
- 3.2 Samples should be collected and stored in polyethylene bottles. Maximum holding time is 28 days.

4. Interferences

- 4.1 Metals, such as mercury, complex ammonia and cause low results.
- 4.2 Substances, mostly metals, which are insoluble in basic solution can cause turbidity interference.
- 4.3 Metals such as manganese, which have two readily available oxidation states, catalyze the indophenol reaction and can enhance the color formation.
- 4.4 The addition of chelating agents such as citrate, EDTA, tartrate, and combinations of these, effectively decomplex the ammonia and complex the metals.
- 4.5 Nitroprusside has been found to stabilize the indophenol reaction and avoid sensitivity variations caused by metals.

5. Apparatus

- 5.1 Technicon BD-40 Block Digestor.
- 5.2 Pyrex Test Tubes, Folin-Wu Digestion Tubes, 25 x 200 mm.
- 5.3 Vortex Genie Mixer.
- 5.4 Technicon #114-0009-02 Rack (Modified).
- 5.5 Sampler IV.
- 5.6 Analytical Cartridge ($\text{NH}_3\text{-N}$) AA II.
- 5.7 Proportioning Pump III.
- 5.8 Heating Bath, 40° C., AA I.
- 5.9 Colorimeter, 15 mm Flow Cell, S10 Phototube, 630 nm Filters.
- 5.10 Recorder.
- 5.11 Sulfuric Acid Trap (for air purification)

6. Reagents

All chemicals are ACS "Reagent" grade and all reagent water is deionized and distilled.

- 6.1 Digestion Solution: Dissolve 2 gm HgO in 25 ml of 6N H_2SO_4 . Add 200 ml of conc. H_2SO_4 to 500 ml of the reagent water. While the strong acid solution is still hot, 134 gm of K_2SO_4 are dissolved in it and then the HgO solution is added. Cool the solution, bring to 1 liter with reagent water and store above 20° C. (No precipitation should occur.)

- 6.2 Dilution Solution: Dilute 6.6 ml of 19 N (50 percent) NaOH to 1 liter with reagent water.
- 6.3 (Replace sodium potassium tartrate solution with the following EDTA reagent.) Disodium ethylenediaminetetraacetate (EDTA), (5 percent): Dissolve 50 g EDTA (disodium salt) and 20 ml 50 percent sodium hydroxide in 1 liter of distilled water.
- 6.4 Alkaline Phenol Solution: Dissolve 80 ml of phenol and 40 ml of 50 percent sodium hydroxide in 800 ml of reagent water, cool, and dilute to 1 liter. Store at 4° C.
- 6.5 Sodium Hypochlorite Solution: Dilute 125 ml of a bleach solution containing 5.25 percent NaOCl to 250 ml with distilled water. Prepare daily!
- 6.6 Sodium Nitroprusside Reagent: Dissolve 0.5 gm of sodium nitroprusside in 900 ml of reagent water and dilute to 1 liter. Store at 4° C.
- 6.7 Quality Control Sample: Solution of nicotinic acid of the desired strength.
- 6.8 Stock Ammonia Solution: Dissolve 3.819 gm of anhydrous ammonium chloride, dried at 105° C, in ammonia free water and dilute to 1 liter. (1 ml = 1 mg NH_3 -N.)
- 6.9 Intermediate Standard: Dilute 100 ml of stock solution (6.8) to 1000 ml with ammonia free water. (1 ml = 0.1 mg NH_3 -N.) Prepare daily.
- 6.10 Working Standards: Prepare daily.

mg/l NH_3 -N

0.1
0.5
2.0
5.0
7.5
10.0

7. Procedure

- 7.1 Place 20 ml of preserved sample into the digestion tube (if the sample is nonhomogeneous, blend in a homogenizer before digestion) and place tube in the digestion rack.
- 7.2 Place 4-8 teflon boiling stones in each tube and 2 ml of digestion solution in each sample.
- 7.3 With each rack of samples, blanks (distilled deionized water), a series of standards, and two quality control samples should be included.

- 7.4 Place the rack of tubes in the block digester and increase the time-temperature settings at the following rate:
- 7.4.1 Evaporate at a block temperature of 200° C. for about 1 1/2 hour.
 - 7.4.2 Increase temperature to 370° C. and digest for about 2 1/2 hours.
- 7.5 Remove the rack of tubes, cool for at least 5 minutes, and add 20 ml of hot reagent water before the samples solidify. Mix samples on a vortex mixer.
- 7.6 The analytical cartridge and reagent tubes are set up according to the schematic (Figure 1).
- 7.7 The colorimeter, recorder, and other equipment are warmed up for approximately 30 minutes with the reagents feeding through the lines.
- 7.8 A baseline is run with all reagents in place and the sampler wash solution feeding through the sample line.
- 7.9 The span of the instrument is synchronized by using the maximum standard and the zero concentration.
- 7.10 The standards are arranged in the sample tray in increasing concentration and the unknown samples, which are digested, are then placed in the sampler tray. Also included in the tray are quality control samples, duplicates, and blanks.
- 7.11 The sample line is switched to the sampler and the analytical run is started.

8. Calculations

- 8.1 The AutoAnalyzers are connected to a computer which receives the response signal from the colorimeter. After the type of curve fit is selected by the operator, the computer calculates the calibration curve by least squares method and generates concentration values for the samples, quality control solution, and laboratory blanks.
- 8.2 The response signal from the colorimeter is also connected to a strip chart recorder. The chart can be used to calculate concentration values by use of the overlay. The standard curve is prepared on the overlay by plotting the peak heights of standards against known concentrations. The concentrations of the samples are obtained by comparing sample peak heights with the standard curve. The standard curve is not linear throughout the working range.

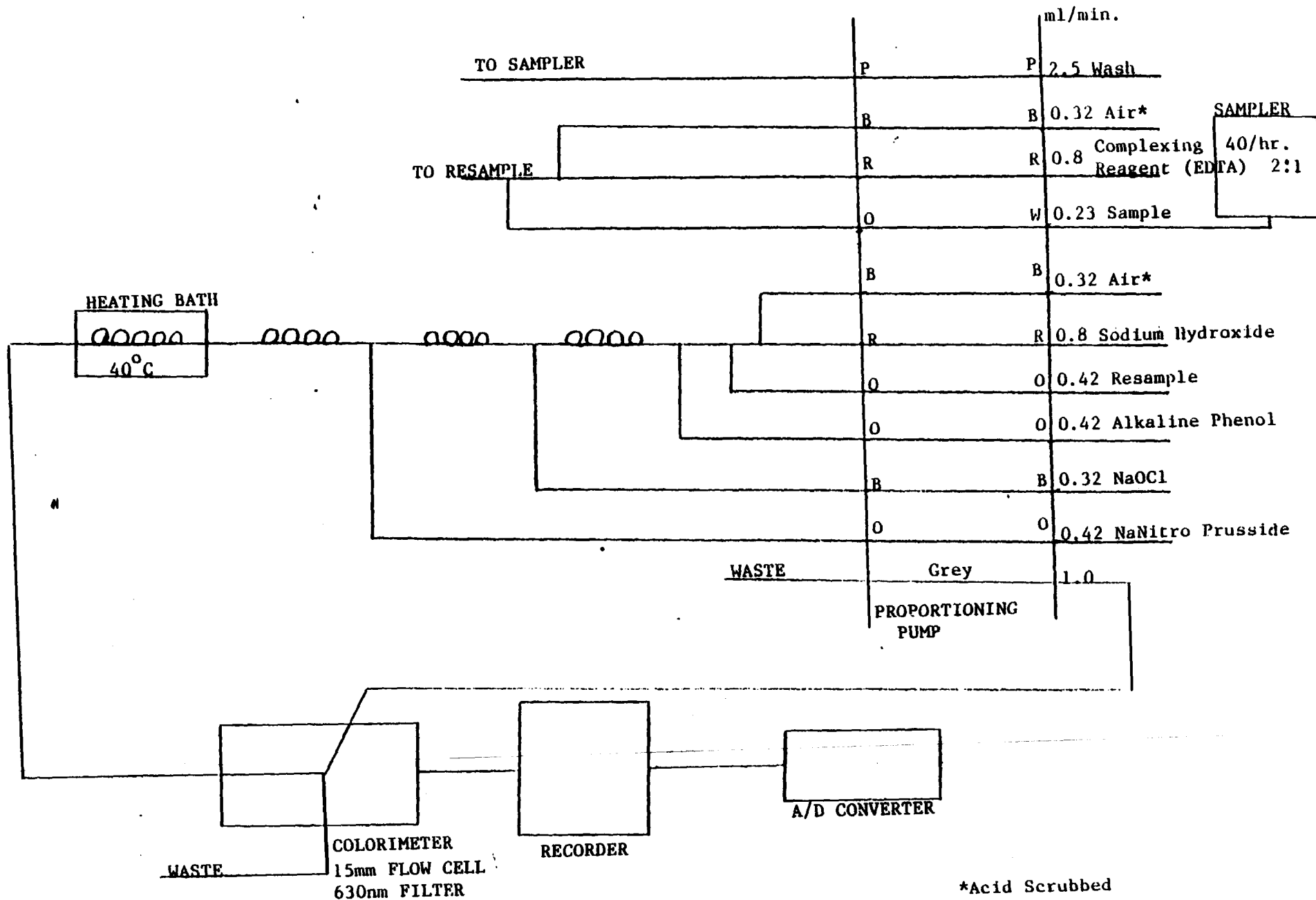
9. Precision and Accuracy

- 9.1 **Detection Limit:** This is defined as three times the standard deviation of the blank as determined by replicate blank analyses and the results of the blank for each run. Our detection limit at present is 0.1 mg/l N.
- 9.2 The precision and accuracy data for this analysis is obtained from the quality controls and real sample duplicates which are run 5-10 percent of the time.
- 9.3 Control limits are calculated at ± 3 standard deviations from the mean value of the quality control standards.
- 9.4 A spiked sample is analyzed for every 20 samples.

10. References

- 10.1 Ultramicro Semiautomated Method for Simultaneous Determination of Total Phosphorus and Total Kjeldahl Nitrogen in Wastewaters, Andrea M. Jirka, Mark Carter, Dorothy May, and Frederic Fuller, Environmental Science and Technology, p. 1038, Vol. 10, No. 10, October, 1976.
- 10.2 Methods for Chemical Analysis of Water and Wastes, EPA, p. 351.2, 1983.

ENV LABS 3
2-4-88
NAC
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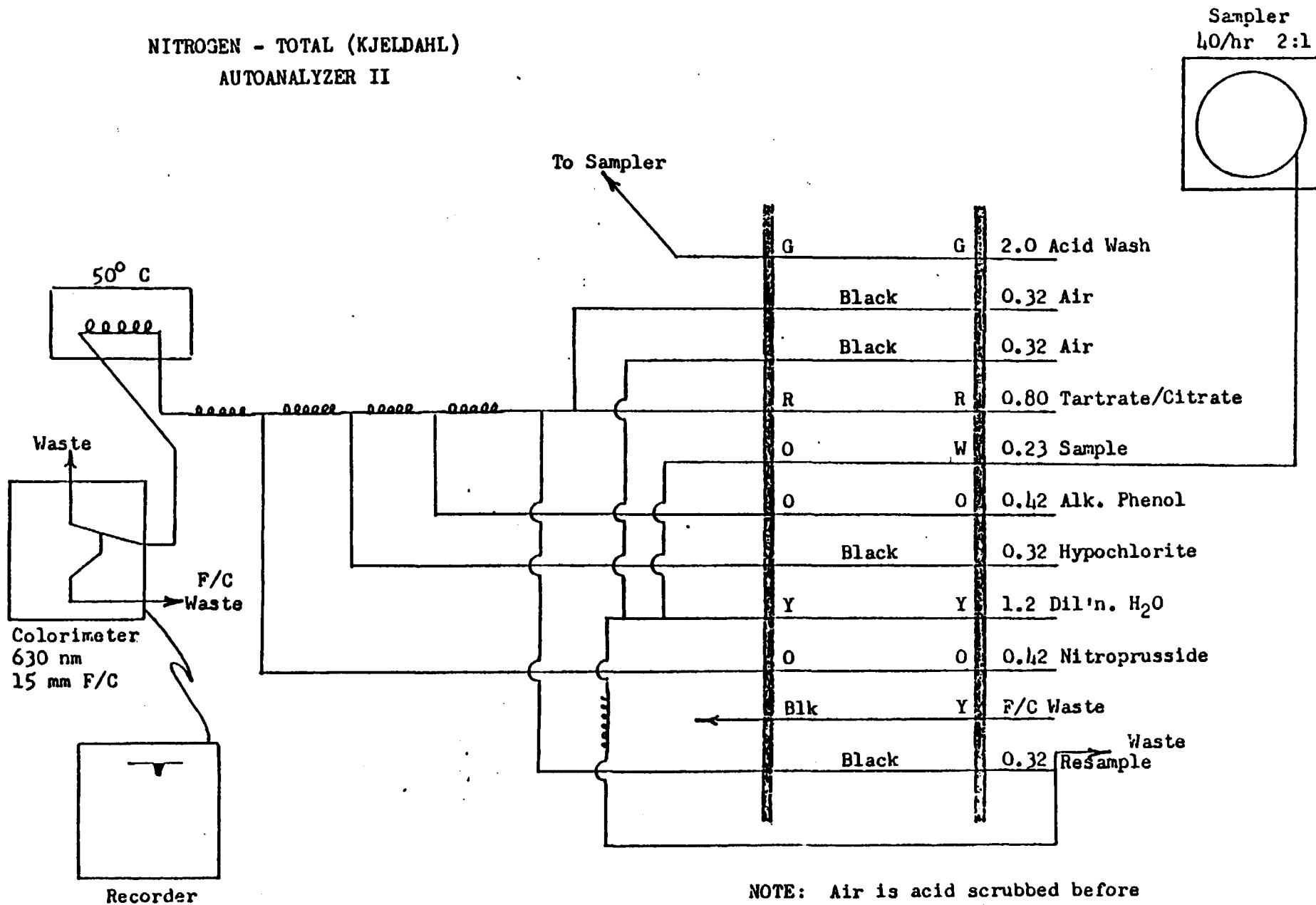
Nitrogen--Total (KJELDAML)

Autoanalyzer II

(Modified 11-15-85)

NITROGEN - TOTAL (KJELDAHL)
AUTOANALYZER II

FIGURE 1



NOTE: Air is acid scrubbed before introduction into system.

PHOSPHORUS, ALL FORMS
(Colorimetric, Automated, Ascorbic Acid)
ISBH Modification, 1983 EPA Manual

ISBH Code No. P-A-1-88
STORET NO. See Section 4
Approved for NPDES

1. Scope and Application

- 1.1 These methods cover the determination of specified forms of phosphorus in drinking, surface and saline waters, domestic and industrial wastes.
- 1.2 The methods are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pre-treatment of the sample, the various forms of phosphorus given in Figure 1 may be determined. These forms are defined in Section 4.
 - 1.2.1 Except for in-depth and detailed studies, the most commonly measured forms are phosphorus and dissolved phosphorus, and orthophosphate and dissolved orthophosphate. Hydrolyzable phosphorus is normally found only in sewage-type samples. Insoluble forms of phosphorus are determined by calculation.
- 1.3 The methods are usable in the 0.03 to 2.0 mg P/l range. Approximately 40 samples per hour can be analyzed.

2. Summary of Method

- 2.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.
- 2.2 Only orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by manual sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by manual persulfate digestion. The developed color is measured automatically on the AutoAnalyzer.

3. Sample Handling and Preservation

- 3.1 If benthic deposits are present in the area being sampled, great care should be taken not to include these deposits.
- 3.2 Sample containers may be of plastic material; such as cubitainers, or of Pyrex glass.

- 3.3 If the analysis cannot be performed the same day of collection, the sample should be preserved by the addition of 2 ml 50 percent H_2SO_4 per liter and refrigeration at 4° C. Maximum holding time is 28 days.

4. Definitions and Storet Numbers

- 4.1 Total Phosphorus (P): All of the phosphorus present in the sample regardless of form, as measured by the persulfate digestion procedure (00665).
- 4.1.1 Total Orthophosphate (P-ortho): Inorganic phosphorus [$(PO_4)^{-3}$] in the sample as measured by the direct colorimetric analysis procedure (70507).
- 4.1.2 Total Hydrolyzable Phosphorus (P-hydro): Phosphorus in the sample as measured by the sulfuric acid hydrolysis procedure, and minus predetermined orthophosphates. This hydrolyzable phosphorus includes polyphosphates $(P_2O_7)^{-4}$, $(P_3O_{10})^{-5}$, etc. plus some organic phosphorus (00669).
- 4.1.3 Total Organic Phosphorus (P-org): Phosphorus (inorganic plus oxidizable organic) in the sample as measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate (00670).
- 4.2 Dissolved Phosphorus (P-D): All of the phosphorus present in the filtrate of a sample filtered through a phosphorus-free filter of 0.45 micron pore size and measured by the persulfate digestion procedure (00666).
- 4.2.1 Dissolved Orthophosphate (P-D): As measured by the direct colorimetric analysis procedure (00671).
- 4.2.2 Dissolved Hydrolyzable Phosphorus (P-D, hydro): As measured by the sulfuric acid hydrolysis procedure and minus predetermined dissolved orthophosphates (00672).
- 4.2.3 Dissolved Organic Phosphorus (P-D, org): As measured by the persulfate digestion procedure, and minus dissolved hydrolyzable phosphorus and orthophosphate (00673).

- 4.3 The following forms, when sufficient amounts of phosphorus are present in the sample to warrant such consideration, may be calculated:

4.3.1 Insoluble Phosphorus (P-I)=(P)-(P-D) (00667).

4.3.1.1 Insoluble orthophosphate
(P-I, ortho)=(P, ortho)-(P-D, ortho)
(00675).

4.3.1.2 Insoluble Hydrolyzable Phosphorus
(P-I, hydro)=(P, hydro)-(P-D, hydro)
(00675).

4.3.1.3 Insoluble Organic Phosphorus (P-I,
org)=(P, org)-(P-D, org) (00676).

- 4.4 All phosphorus forms shall be reported as P, mg/l, to the second place.

5. Interferences

- 5.1 No interference is caused by copper, iron, or silicate at concentrations many times greater than their reported concentration in sea water. However, high iron concentrations can cause precipitation of and subsequent loss of phosphorus.
- 5.2 The salt error for samples ranging from 5 to 20 percent salt content was found to be less than 1 percent.
- 5.3 Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus. However, at concentrations found in sea water, it does not interfere.
- 5.4 Sample turbidity must be removed by filtration prior to analysis for orthophosphate. Samples for total or total hydrolyzable phosphorus should be filtered only after digestion. Sample color that absorbs in the photometric range used for analysis will also interfere.

6. Apparatus

- 6.1 Technicon AutoAnalyzer consisting of:
- 6.1.1 Sampler.
- 6.1.2 Analytical Cartridge (AAII).
- 6.1.3 Proportioning pump.
- 6.1.4 Heating bath, 37° C.

- 6.1.5 Colorimeter equipped with 15 or 50 mm tubular flow cell.
- 6.1.6 660 nm filter.
- 6.1.7 Recorder.
- 6.1.8 A/D Converter.
- 6.2 Autoclave.
- 6.3 Acid-washed glassware: All glassware used in the determination should be washed with 1:1 HCl and rinsed with distilled water. The acid-washed glassware should be filled with distilled water and treated with all the reagents to remove the last traces of phosphorus that might be absorbed on the glassware. Preferably, this glassware should be used only for the determination of phosphorus and after use it should be rinsed with distilled water and kept covered until needed again. If this is done, the treatment with 1:1 HCl and reagents is only required occasionally. Commercial detergent should never be used.

7. Reagents

- 7.1 Sulfuric acid solution, 5 N: Slowly add 70 ml of conc. H_2SO_4 to approximately 400 ml of distilled water. Cool to room temperature and dilute to 500 ml with distilled water.
- 7.2 Antimony potassium tartrate solution: Weigh 0.3 g $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$, dissolve in 50 ml distilled water in 100 ml volumetric flask, dilute to volume. Store at 4° C.
- 7.4 Ascorbic acid, 0.1 M: Dissolve 1.8 g of ascorbic acid in 100 ml of distilled water. (Make fresh daily.)
- 7.5 Combined reagent: Mix the above reagents in the following proportions for 100 ml of the mixed reagent: 50 ml of 5 N H_2SO_4 (7.1), 5 ml of antimony potassium tartrate solution (7.2), 15 ml of ammonium molybdate solution (7.3), and 30 ml of ascorbic acid solution (7.4). Mix after addition of each reagent. All reagents must reach room temperature before they are mixed and must be mixed in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until the turbidity disappears before processing. This volume is sufficient for 4 hours operation. Since the stability of this solution is limited, it must be freshly prepared for each run.

Note: A stable solution can be prepared by not including the ascorbic acid in the combined reagent. If this is done, the mixed reagent (molybdate, tartrate, and acid) is pumped through the distilled water line and the ascorbic acid solution (30 ml of 7.4 diluted to 100 ml with distilled water) through the original mixed reagent line.

- 7.6 Sulfuric acid solution, 11 N: Slowly add 310 ml conc. H_2SO_4 to 600 ml distilled water. When cool, dilute to 1 liter.
- 7.7 Ammonium persulfate.
- 7.8 Acid wash water: Add 40 ml of sulfuric acid solution (7.6) to 1 liter of distilled water and dilute to 2 liters. (Not to be used when only orthophosphate is being determined.)
- 7.9 Phenolphthalein indicator solution (5 g/l): Dissolve 0.5 g of phenolphthalein in a solution of 50 ml of ethyl or isopropyl alcohol and 50 ml of distilled water.
- 7.10 Stock phosphorus solution: Dissolve 0.4393 g of pre-dried ($150^\circ C.$ for 1 hour) KH_2PO_4 in distilled water and dilute to 1000 ml. 1 ml = 0.1 mg P.
- 7.11 Standard phosphorus solution: Dilute 50 ml of stock solution (7.10) to 1000 ml with distilled water. 1 ml = .005 mg P.
- 7.12 Prepare a series of standards by diluting suitable volumes of standard solutions to 200 ml with distilled water. The following dilutions are suggested:

<u>ml of Standard Phosphorus Solution</u>	<u>mg P/l</u>
20 ml of 0.3 ppm	0.03
20 ml of 0.5 ppm	0.05
20 ml of 1.0 ppm	0.1
40 ml of 1.5 ppm	0.3
20 ml of 5.0 ppm	0.5
40 ml of 5.0 ppm	1.0
60 ml of 5.0 ppm	1.5

- 7.13 Sodium chloride solution: Dissolve 20 g NaCl and 4 drops of Levor V in 1 liter of distilled water.

8. Procedure

8.1 Phosphorus

- 8.1.1 Add 0.5 ml of sulfuric acid solution (7.6) to a 30 ml sample in a 25 x 150 mm culture tube.
- 8.1.2 Add 0.4 g of ammonium persulfate.
- 8.1.3 Heat for 20 minutes in an autoclave at 121° C. (15-20 psi).
- 8.1.4 Determine phosphorus as outlined in (8.3.2) with acid wash water (7.8) in wash tubes.

8.2 Hydrolyzable Phosphorus

- 8.2.1 Add 0.5 ml of sulfuric acid solution (7.6) to a 30 ml sample in a 25 x 150 mm culture tube.
- 8.2.2 Heat for 30 minutes in an autoclave at 121° C. (15-20 psi).
- 8.2.3 Cool and dilute the sample to 50 ml. If sample is not clear at this point, filter.
- 8.2.4 Determine phosphorus as outlined in (8.3.2) with acid wash water (7.8) in wash tubes.

8.3 Orthophosphate

- 8.3.1 Add 1 drop of phenolphthalein indicator solution (7.9) to approximately 50 ml of sample. If a red color develops, add sulfuric acid solution (7.6) drop-wise to just discharge the color. Acid samples must be neutralized with 1 N sodium hydroxide (40 g NaOH/l).
- 8.3.2 Set up manifold as shown in Figure 1 AA II.
- 8.3.3 Allow both colorimeter and recorder to warm up for 30 minutes. Obtain a stable baseline with all reagents, feeding distilled water through the sample line.
- 8.3.4 For the AA II system, use a 40/hr, 2:1 cam, and a common wash.
- 8.3.5 Place standards in sampler in order of decreasing concentration. Complete filling of sampler tray with unknown samples.
- 8.3.6 Switch sample line from distilled water to sampler and begin analysis.

9. Calculation

- 9.1 Prepare a standard curve by plotting peak heights of processed standards against known concentrations. Compute concentrations of samples by comparing sample peak heights with standard curve. Any sample whose computed value is less than 5 percent of its immediate predecessor must be rerun.
- 9.2 Data reduction is also done on computer support equipment.

10. Quality Control

- 10.1 The following are analyzed for every 20 samples:

1. Quality control sample.
2. Duplicate
3. Spiked sample.
4. Blanks.

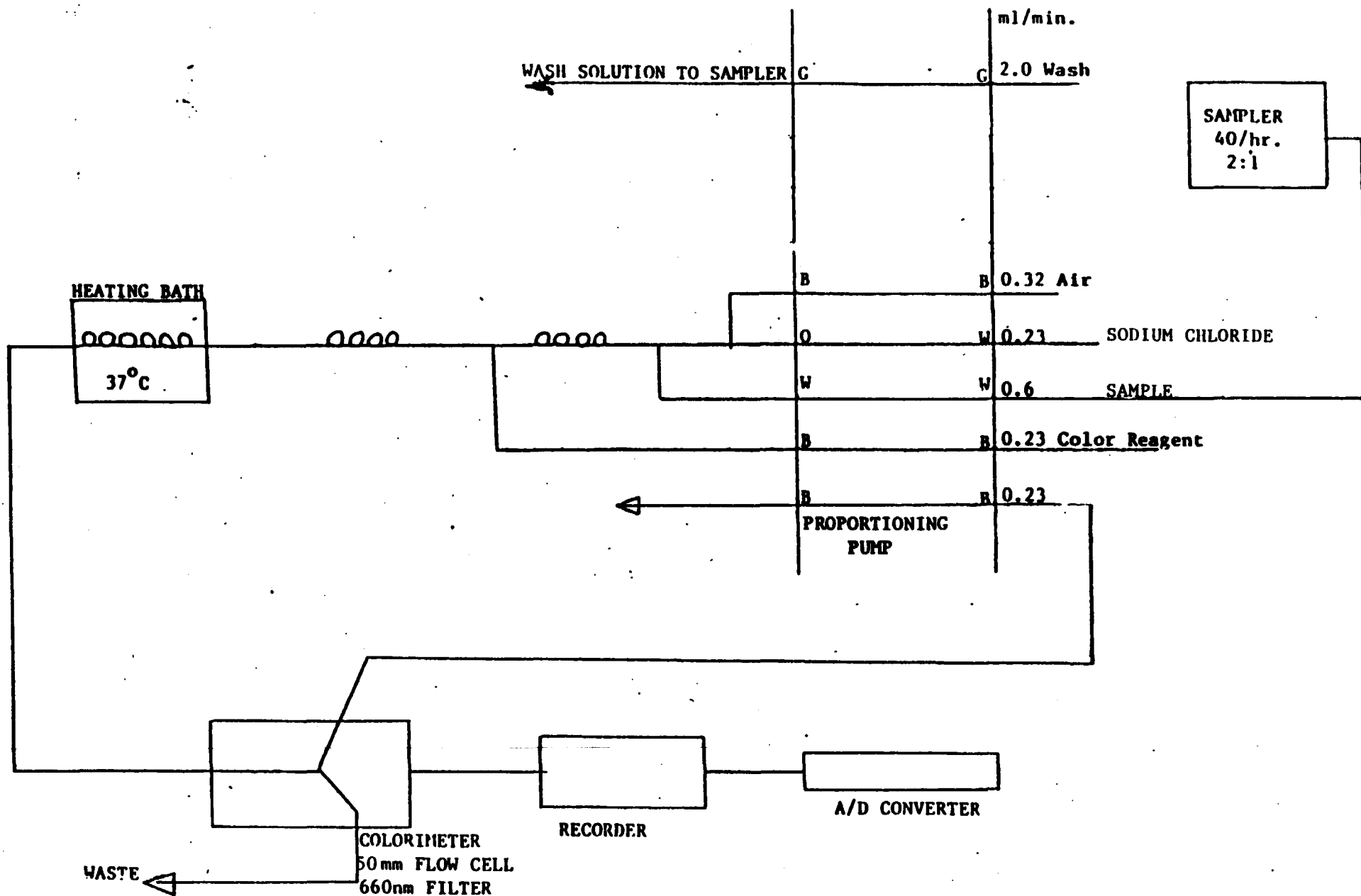
- 10.2 Internal audits using USEPA ampules.

- 10.3 Interlaboratory studies sponsored by the USEPA.

11. References

- 11.1 Murphy, J., and Riley, J., A Modified Single Solution for the Determination of Phosphate in Natural Waters, Anal. Chem. Acta., 27, 31, 1962.
- 11.2 Gales, M., Jr., Julian, E., and Kroner, R., Method for Quantitative Determination of Total Phosphorus in Water, Jour AWWA, 58, No. 10, 1363.
- 11.3 Lobring, L. B., and Booth, R. L., Evaluation of the AutoAnalyzer II; A Progress Report, Technicon International Symposium, June 1972, New York, NY.
- 11.4 Annual book of ASTM Standards, Part 31, Water, Standard D515-72, p. 388, 1976.
- 11.5 Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 450, Method 424G, 1975.
- 11.6 Methods for Chemical Analysis of Water and Wastes, p. 365.1, 1983.

ENCLOSURE 3
2-4-88
NAC
INCL. TAT



PHOSPHORUS MANIFOLD

AUTOANALYZER II

(MODIFIED 5-15-88)

SOLIDS, FILTERABLE (DISSOLVED)
(EPA Method, 1983)

ISBH Code No. SG-A-1-88
STORET No. 70300 (180° C.)
00515 (105° C.)

Approved for NPDES

1. Scope and Application

- 1.1 This method is applicable to surface waters, domestic and industrial wastes, and saline waters.
- 1.2 The practical range of the determination is 10 mg/l to 20,000 mg/l.

2. Summary of Method

- 2.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180° C.

3. Definitions

- 3.1 Filterable solids are defined as those solids capable of passing through a standard glass fiber filter and dried to constant weight at 180° C.

4. Sample Handling and Preservation

- 4.1 Samples should be analyzed as soon as practicable. Maximum holding time is 48 hours.

5. Interferences

- 5.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying and desiccation and quick weighing.
- 5.2 Samples containing high concentrations of bicarbonate will require careful and possible prolonged drying at 180° C. to insure that all the bicarbonate is converted to carbonate.
- 5.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

6. Apparatus

- 6.1 Glass fiber filter, 4.7 cm or 2.2 cm, without organic binder, Reeve Angel type 984 H, Gelman type A, or equivalent.
- 6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.
- 6.3 Suction flask, 500 ml.
- 6.4 Gooch crucibles, 25 ml (if 2.2 cm filter is used).
- 6.5 Evaporating dishes, porcelain, 100 ml volume. (Vycor or platinum dishes may be substituted).
- 6.6 Steam bath.
- 6.7 Drying oven, $180^{\circ} \text{C} \pm 2^{\circ} \text{C}$.
- 6.8 Desiccator.
- 6.9 Analytical balance, 200 g capacity, capable of weighing to 0.1 mg.

7. Procedure

- 7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through.
- 7.2 Preparation of evaporating dishes: Heat the clean dish to 550°C . for one hour in a muffle furnace. Cool in desiccator and store until needed. Weigh immediately before use.
- 7.3 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml volumetric cylinder. If suspended matter is low, a larger volume may be filtered.
- 7.4 Filter the sample through the glass fiber filter and continue to apply vacuum for about three minutes after filtration is complete to remove as much water as possible.
- 7.5 Transfer 100 ml (or a larger volume) of the filtrate to a weighed evaporating dish and evaporate to dryness on a steam bath.
- 7.6 Dry the evaporated sample for at least one hour at $180^{\circ} \text{C} \pm 2^{\circ} \text{C}$. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

- 7.7 Note: The filtrate from the test for SOLIDS, NONFILTERABLE, may be used for this determination.

8. Calculation

- 8.1 Calculate filterable solids as follows:

$$\text{Filterable solids, mg/l} = \frac{(\text{Wt. of dried residue + dish}) - (\text{wt. of dish}) \times 1000}{\text{Volume of filtrate used}}$$

9. Quality Control

- 9.1 The following are analyzed for every twenty (20) samples:

1. Quality control sample.
2. Duplicate.
3. Blank.

- 9.2 Internal audits using USEPA ampules.

- 9.3 Interlaboratory studies sponsored by USEPA.

10. References

- 10.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 95, Method 209B, 1985.
- 10.2 Methods for Chemical Analysis of Water and Wastes, 1983 Environmental Protection Agency, p. 160.1.
- 10.3 Federal Register, Vol. 38, No. 199, (October 16, 1973), Part II, EPA, Water Programs.

ENV LABS 2
2-3-88
NAE
SOLIDS 2. TXT

SOLIDS, NONFILTERABLE (SUSPENDED)
(EPA Method, 1983)

ISBH Code No. SNF-A-1-88
STORET No. 00530
Approved for NPDES

1. Scope and Application

- 1.1 This method is applicable to surface waters, domestic and industrial wastes, and saline waters.
- 1.2 The practical range of the determination is 10 mg/l to 20,000 mg/l.

2. Summary of Method

- 2.1 A well-mixed sample is filtered through a standard glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105° C.

3. Definitions

- 3.1 Nonfilterable solids are defined as those solids which are retained by a standard glass fiber filter and dried to constant weight at 103-105° C.

4. Sample Handling and Preservation

- 4.1 Nonhomogeneous particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample.
- 4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Maximum holding time is seven days.

5. Interferences

- 5.1 Too much residue on the filter will entrap water and may require prolonged drying.

6. Apparatus

- 6.1 Glass fiber filter discs, 4.7 cm or 2.2 cm, without organic binder, Reeve Angel type 984 H, Gelman type A, or equivalent.
- 6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.
- 6.3 Suction flask, 500 ml.
- 6.4 Gooch crucibles, 25 ml (if 2.2 cm filter is used).
- 6.5 Drying oven, 103-105° C.

- 6.6 Desiccator.
- 6.7 Analytical balance, 200 g capacity, capable of weighing to 0.1 mg.

7. Procedure

- 7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in a muffle furnace at 550° C. for one hour. Remove to desiccator and store until needed. Weigh immediately before use.
- 7.2 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml volumetric cylinder. If suspended matter is low, a larger volume may be filtered.
- 7.3 Carefully remove the filter from the membrane filter assembly. Alternatively, remove crucible and filter from crucible adapter. Place in drying oven and dry at 103-105° C. to constant weight.

8. Calculations

- 8.1 Calculate nonfilterable solids as follows:

$$\text{Nonfilterable Solids (mg/l)} = \frac{(\text{mg}) - (\text{mg})}{\text{ml of sample filtered}} \times 1000$$

(mg) (mg)

(Wt. of filter + residue) - (wt. of filter)

9. Quality Control

- 9.1 Blanks and duplicates are analyzed for every twenty (20) samples.
- 9.2 Internal audits using USEPA ampules.
- 9.3 Interlaboratory studies sponsored by USEPA.

10. References

- 10.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 96, Method 209C, 1985.
- 10.2 Methods for Chemical Analysis of Water and Wastes, 1983 Environmental Protection Agency, p. 160.2.
- 10.3 Federal Register, Vol. 38, No. 199, (October 16, 1973), Part II, EPA, Water Programs.

ENV LABS 2
2-3-88
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SOLIDS.TXT

SOLIDS, VOLATILE
(EPA Method, 1983)

ISBH Code No. SV-A-4-87
STORET No. 00505 (ST)
00520 (SF)
00535 (SNF)
Approved for NPDES

1. Scope and Application

- 1.1 This method determines the weight of solid material combustible at 550 Deg. C.
- 1.2 The test is useful in obtaining a rough approximation of the amount of organic matter present in the solid fraction of sewage, activated sludge, industrial wastes, or bottom sediments.

2. Summary of Method

- 2.1 The residue obtained from the determination of total, suspended, or dissolved solids is ignited at 550 Deg. C. in a muffle furnace. The loss of weight on ignition is reported as mg/l volatile solids.

3. Comments

- 3.1 The test is subject to many errors due to loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion.
- 3.2 The results should not be considered an accurate measure of organic carbon in the sample, but may be useful in the control of plant operations.
- 3.3 The principal source of error in the determination is failure to obtain a representative sample.

4. Quality Control

- 4.1 The following are the quality controls performed :

1. Duplicates
2. Blanks

5. References

- 5.1 Standard Methods for the Examination of Water and Wastewater, 16th Ed., p. 97, Method 209D.
- 5.2 Methods for Chemical Analysis of Water and Wastes, 1983, Environmental Protection Agency, p. 180.4.
- 5.3 Federal Register, Vol. 48, No. 109, October 26, 1983, Part VIII, EPA, Water Programs.

SULFATE
(Methylthymol Blue Automated Method)
(16th Edition "Standard Methods")

ISBH Code No. SO₄-B-1-88
STORET No. 00945⁴
Approved for NPDES

1. Scope and Application

- 1.1 This method is applicable to potable, surface, and saline waters as well as domestic and industrial wastes.
- 1.2 The method is suitable for a range of 5 - 100 mg/l SO₄. This range can be modified by making changes in the sulfate manifold. Approximately 40 samples per hour can be analyzed.

2. Summary of Method

- 2.1 In this method for determining sulfate, it is necessary to remove interference by passing the sample through a cation-exchange column. The sample containing sulfate is then reacted with barium chloride to form barium sulfate at a pH of 2.5 to 3.0. Excess barium reacts with methylthymol blue to form a blue-colored chelate at a pH of 12.5 to 13.0. The uncomplexed methylthymol blue is gray in color, and when it is chelated with barium it forms a blue color. Initially the barium chloride and methylthymol blue are present in equimolar amounts. Therefore, the amount of uncomplexed methylthymol blue is equal to the sulfate present.

3. Sampling and Handling

- 3.1 No preservative is needed.
- 3.2 Samples are collected in polyethylene bottles.
- 3.3 Samples should be stored at low temperature (4° C.).
- 3.4 Maximum holding time is 28 days.

4. Interference

- 4.1 Color, turbidity, cations such as calcium, aluminum, and iron interfere, but are removed by the cation-exchange column.

5. Apparatus

- 5.1 Technicon AutoAnalyzer consisting of:
 - 5.1.1 Sampler I with 40/hr - 1:1 cam.
 - 5.1.2 Sulfate Manifold.
 - 5.1.3 Proportioning Pump.
 - 5.1.4 Colorimeter equipped with 50 mm tubular flow-cell and 460 nm filters.
 - 5.1.5 Recorder.
 - 5.1.6 A/D Converter.

6. Reagents

- 6.1 Barium Chloride Solution: Dissolve 1.526 gm $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in 900 ml of distilled water and dilute to 1 liter. Store in a polyethylene bottle.
- 6.2 Hydrochloric Acid, 1.0 N Solution: Add 83 ml of conc. HCl to 800 ml of distilled water and dilute to 1 liter.
- 6.3 Methylthymol Blue solution: Dissolve 0.1182 gm of methylthymol blue in 25 ml of barium chloride solution (6.1). Add 4 ml of 1.0 N HCl solution (6.2), which produces a bright orange color. Add 71 ml of distilled water and dilute to 500 ml with ethanol (95 percent reagent grade). The pH of this solution should be 2.6. Store in a brown glass bottle in the refrigerator and prepare weekly.
- 6.4 Buffered EDTA Solution: Dissolve 6.75 gm NH_4Cl in 500 ml of distilled water. Add 75 ml of conc. NH_4OH and dilute to 1 liter with distilled water. Add and dissolve 40 gm of tetrasodium EDTA. Store in a polyethylene bottle.
- 6.5 Sodium Hydroxide, 0.18 N Solution: Add 12 ml of 50 percent NaOH to 800 ml of distilled water and dilute to 1 liter. Prepare fresh weekly.
- 6.6 Stock Sulfate Solution, 100 mg/l: Dissolve 1.479 gm of anhydrous Na_2SO_4 in 500 ml of distilled water and dilute to 1 liter.

- 6.6.1 Prepare a series of working standards by diluting volumes of stock solution to 200 ml with distilled water. The following concentrations are suggested:

mg/l SO₄

5.
10.
20.
40.
60.
80.
100.

- 6.7 Ion-exchange Column: The column is made of a length of glass tubing 7.5 inches long x 2.0 mm ID x 3.6 mm OD. Wash the cation-exchange resin three times with distilled water to remove the fines. Next fill the column with the resin, being careful not to allow air to become trapped in the column. Place glass wool plugs in each end to prevent resin from escaping. Use Bio Rex 70, 20-50 mesh, Na⁺ form.

- 6.8 Dilution Water: Distilled water.

7. Procedure

- 7.1 No advance sample preparation is required. Set up the manifold as shown in the schematic (Figure 1).
- 7.2 Allow the colorimeter and recorder to warm up for 30 minutes.
- 7.3 Run a baseline with all reagents, feeding distilled water through the sample line, then place the cation-exchange column in place. Adjust the colorimeter to obtain a stable baseline and set the span on the recorder to obtain the working range.
- 7.4 Sample at the rate indicated on the schematic.
- 7.5 Place the working standards in the sampler tray in increasing order of sulfate concentration. Complete filling the sampler tray with unknown samples.
- 7.6 Run at least two quality control samples and two duplicate samples in each tray.
- 7.7 Start the sample run once a stable baseline is obtained.
- 7.8 At the end of the sample run the system should be purged with a solution of buffered EDTA. This can be done by placing the methylthymol blue line and the NaOH line in water for a few minutes and then into the EDTA for ten

minutes. Then wash with water for fifteen minutes before shutting down. Remove the resin column while full of water if it is to be used again. Rinsing also with 1.0 N HCl in the same manner as EDTA aids in removal of build-up in the flow-cell.

8. Calculations

- 8.1 The AutoAnalyzers are connected to a computer which receives the response signal from the colorimeter. After the type of curve fit is selected by the operator, the computer calculates the calibration curve by least squares method and generates concentration values for the samples, quality control solution, and laboratory blanks.
- 8.2 The response signal from the colorimeter is also connected to a strip chart recorder. The chart can be used to calculate concentration values by use of the overlay. The standard curve is prepared on the overlay by plotting the peak heights of standards against known concentrations. The concentration of the samples are obtained by comparing sample peak heights with the standard curve. The standard curve is not linear throughout the working range.

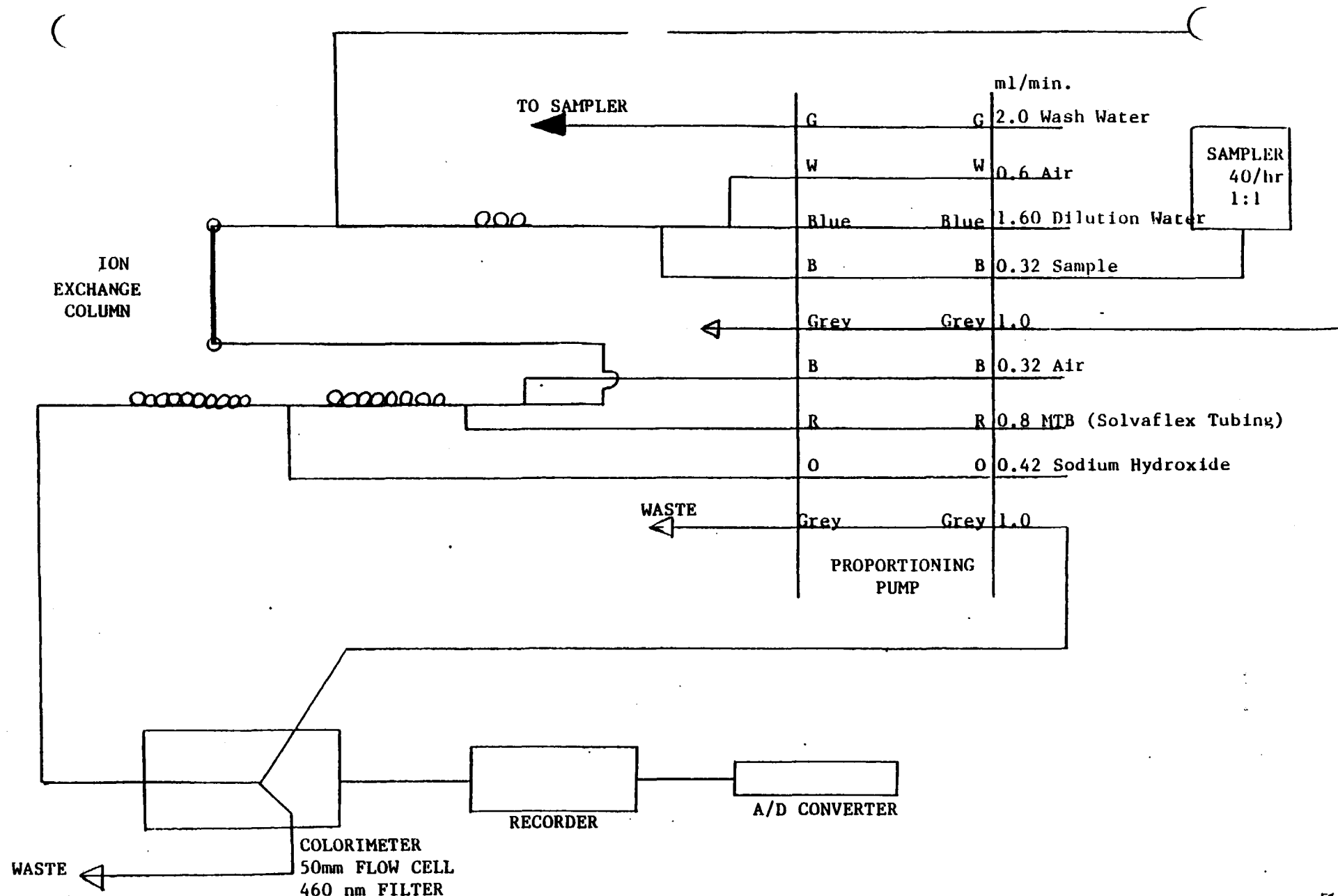
9. Quality Control

- 9.1 The following are analyzed for every 20 samples:
1. Quality control sample.
 2. Duplicate.
 3. Spiked sample.
 4. Blanks.
- 9.2 Internal audits using USEPA ampules.
- 9.3 Interlaboratory studies sponsored by the USEPA.

10. References

- 10.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, p 468, Method 426D, 1985.
- 10.2 Technicon Industrial Systems. Sulfate in Water and Wastes, (Industrial Method AA II 118-71W), December 1972.

ENV LABS 3
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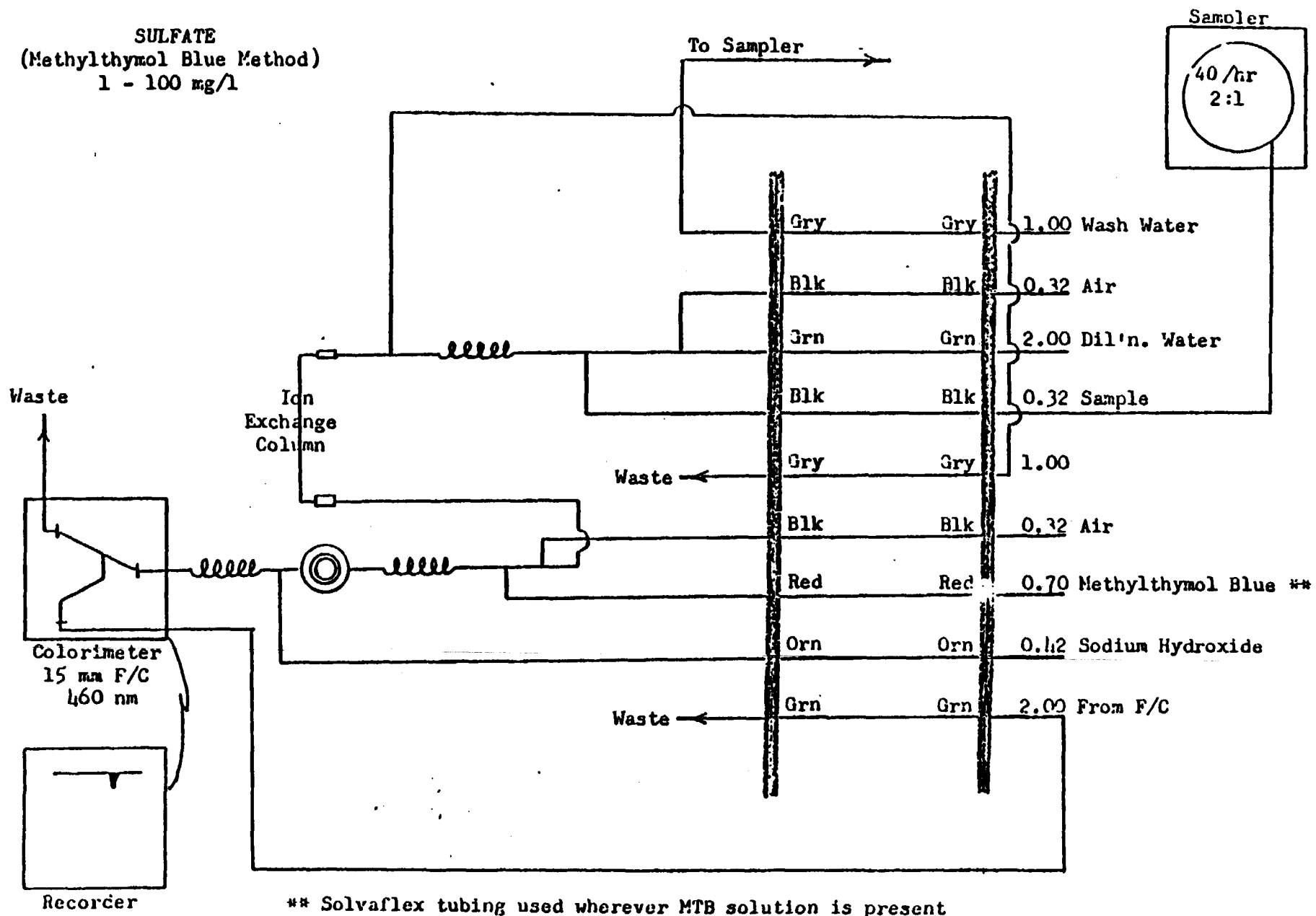
SULFATE MANIFOLD
(METHYL THYMOL BLUE METHOD)

AUTOANALYZER II

(MODIFIED 11-19-85)

SULFATE
(Methylthymol Blue Method)
1 - 100 mg/l

Figure 1



Appendix E
FIELD TESTING PROCEDURES

GLT821/15-4

FIELD MEASUREMENT OF pH

METHOD: Electrometric

REFERENCE: Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Method 150.1, 1983

SENSITIVITY: 0.1 pH unit

RANGE: 1 to 12 pH units

SAMPLE HOLDING TIME: Less than 6 hours

REAGENTS

- o pH buffer solutions for pH 4, 7, and 10
- o Deionized water in squirt bottle

APPARATUS

- o pH meter
- o Combination electrodes
- o Beakers
- o Glassware that has been washed with soap and water, rinsed twice with hot water, and rinsed twice with deionized water

CALIBRATION

1. Place electrode in pH 7 buffer solution.
2. Allow meter to stabilize and then turn calibration dial until a reading of 7.0 is obtained.
3. Rinse electrode with deionized water and place it in a pH 4 or pH 10 buffer solution.
4. Allow meter to stabilize again and then turn slope adjustment dial until a reading of 4.0 is obtained for the pH 4 buffer solution or 10.0 for the 10 pH solution.

5. Rinse electrode with deionized water and place in pH 7 buffer. If meter reading is not 7.0, repeat Steps 2 to 5.

PROCEDURE

1. Before going into the field:
 - a) Check batteries.
 - b) Do a quick calibration at pH 7 and 4 to check electrode.
 - c) Obtain fresh solutions.
2. Calibrate meter using calibration procedure.
3. Pour the sample into a clean beaker.
4. Rinse electrode with deionized water between samples.
5. Immerse electrode in solution. Make sure the white KCl junction on side of electrode is in the solution. The level of electrode solution should be one inch above sample to be measured.
6. Recheck calibration with pH 7 buffer solution after every five samples.

When calibrating the meter, use pH buffers 4 and 7 for samples with pH < 8, and buffers 7 and 10 for samples with pH > 8. If meter will not read pH 4 or 10, something may be wrong with the electrode. Return it to the laboratory and explain the malfunction.

Measurement of pH is temperature dependent. Therefore, temperatures of buffers and samples should be within about 2°C. For refrigerated or cool samples, use refrigerated buffers to calibrate the pH meter.

Weak organic and inorganic salts and oil and grease interfere with pH measurements. If oil and grease are visible, note on data sheet. Clean electrode with soap and water and rinse with a 10 percent solution of HCl. Then recalibrate meter.

Following field measurements:

- a) Report any problems.
- b) Compare with previous data.
- c) Clean all dirt off meter and inside case.
- d) Store electrode in pH 4 buffer.

ACCURACY AND PRECISION

Accuracy and precision are dependent on the instrument used;
refer to manufacturer's manual

Expected accuracy and precision are ± 0.1 pH unit.

PREVENTATIVE MAINTENANCE

Refer to manufacturer's manual.

FIELD MEASUREMENT OF SPECIFIC CONDUCTANCE
AND TEMPERATURE

METHOD: Specific Conductivity

REFERENCE: Methods for Chemical Analysis of
Water and Wastes, U.S. EPA Method 120.1, 1983

DETECTION
LIMIT: 1 umho/cm @ 25°C

RANGE: 0.1 to 100,000 umho/cm

SAMPLE
HOLDING TIME: Determine onsite or within 24 hours

REAGENTS: Distilled water in squirt bottle and standard
potassium chloride solution

APPARATUS: Conductivity meter and electrode

REAGENT PREPARATION

1. Stock Potassium Chloride (KCl) Solution (1.00 N): Dissolve 74.555 g KCl in distilled water and dilute to 1,000 ml in a volumetric flask.
2. Standard Potassium Chloride Solution (0.01 N): Dilute 10.0 ml of stock 1.00 N KCl solution to 1,000 ml with distilled water using a volumetric pipet and flask.

PROCEDURE

1. With mode switch at OFF position, check meter zero. If not zeroed, use meter screw to adjust to zero.
2. Plug probe into jack on side of meter.
3. Turn mode switch to red line and turn red line knob until needle aligns with red line on dial. If they cannot be aligned, change the batteries.
4. Immerse probe in 0.01 N standard potassium chloride solution. Do not allow the probe to touch the sample container.

5. Set the mode control to TEMPERATURE. Record the temperature on the bottom scale of the meter in degrees Celsius.
6. Turn MODE switch to appropriate conductivity scale, (i.e., X100, X10, or X1). Use a scale that will give a mid-range output on the meter.
7. Wait for needle to stabilize (about 15 seconds), multiply reading by scale setting, and record the conductivity. The conductivity reading must then be corrected for temperature.

Calculate conductivity using the formula:

$$G_{25} = \frac{G_T}{[1 + 0.02 (T-25)]}$$

where:

G_{25} = conductivity at 25°C, umho/cm

T = temperature of sample, °C

G_T = conductivity of sample at temperature T , umho/cm

Table 1 lists the values of conductivity this solution would have if the distilled water were nonconductive. However, since even high purity distilled water is slightly conductive, the measured conductivity will be higher by an amount equal to the water's conductivity.

Table 1
CONDUCTIVITY METER CALIBRATION TABLE

<u>Temperature (°C)</u>	<u>Conductivity (umho/cm)</u>
15	1,141.5
16	1,167.5
17	1,193.6
18	1,219.9
19	1,246.4
20	1,273.0
21	1,299.7
22	1,326.6
23	1,353.6
24	1,380.8
25	1,408.1
26	1,436.5
27	1,463.2
28	1,490.9
29	1,518.7
30	1,546.7

8. Report results for the standard solution with each data set.
9. Record the type of meter and probe used on field sheet.
10. Rinse probe with deionized water.
11. Wipe meter clean as necessary.
12. Repeat steps 5 through 11 for water samples.
13. After returning to laboratory, compare results with previous data. Report problems to laboratory personnel.

ACCURACY AND PRECISION

Accuracy and precision are dependent on the instrument used. Refer to the manufacturer's manual.

For the YSI Model 33 conductivity meter (per operating manual):

Conductivity

Expected accuracy ± 2.5 percent at 500, 5,000, and 50,000 plus ± 2 percent for probe.

Expected accuracy ± 3 percent at 250, 2,500, and 25,000 plus ± 2 percent for probe.

Temperature

Expected accuracy $\pm 0.1^{\circ}\text{C}$ at 2°C plus probe
 $\pm 0.6^{\circ}\text{C}$ at 45°C plus probe

Probe $\pm 0.1^{\circ}\text{C}$ at 0°C
 $\pm 0.3^{\circ}\text{C}$ at 40°C

PREVENTATIVE MAINTENANCE

Refer to manufacturer's manual.

OVA MONITORING

OPERATION

For complete operating instructions, refer to the manufacturer's instruction manual.

CALIBRATION

By analyzing a gas of known concentration, the OVA is easily calibrated. Methane in air at a concentration of 100 ppm is typically used as the calibration mixture, although the OVA can be calibrated to many other compounds.

Primary calibration of an OVA is performed at the factory.

CALIBRATION PROCEDURE

To calibrate an OVA in the field:

1. Zero the instrument.
2. Sample the calibration mixture and adjust to proper reading. Handle gas cylinders with care.
3. Next, set the calibration switch to a different range.
4. Sample another calibration mixture of different concentration and adjust to proper reading.
5. Zero the instrument.

PREVENTATIVE MAINTENANCE

Refer to manufacturer's instruction manual.

QUALITY CONTROL REQUIREMENTS

Calibrated daily and precision of ± 30 percent.

HNU MONITORING

OPERATION

For complete operating instructions, refer to the manufacturer's instruction manual.

CALIBRATION

By analyzing a gas of known concentration, the HNU is easily calibrated. Benzene is typically used as the calibration gas. When calibrating the HNU, always remember to:

- o Calibrate in the range to be tested.
- o Deliver the calibration gas at ambient temperature and pressure. Handle gas cylinders with care.
- o Calibrate every day.

The calibration gas must be stable during the period of use and must be at a concentration that reflects field sample concentrations. All gas cylinders must have proper regulators.

CALIBRATION PROCEDURE

To calibrate an HNU:

1. Identify the probe by lamp label.
2. Connect the probe.
3. Affirm the ionization potential of the calibration gas.
4. Perform a battery check.
5. Zero the HNU.
6. Sample calibration gas and adjust to proper reading.
7. Repeat steps 5 and 6.
8. If calibration cannot be achieved, the lamp must be cleaned.
9. Replace lamp if the lamp output is too low or if the lamp has failed.

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To obtain more than a two-point calibration, dilute the calibration gas to known concentrations and take additional readings.

PREVENTATIVE MAINTENANCE

Refer to manufacturer's instruction manual.

QUALITY CONTROL REQUIREMENTS

Calibrated daily and precision of ± 30 percent.

GLT808/58

Appendix F
INSTRUCTIONS FOR FILLING OUT SAMPLE DOCUMENTATION

GLT821/15-6

Appendix F
INSTRUCTIONS FOR FILLING OUT SAMPLE DOCUMENTATION

All samples collected at Superfund sites for laboratory analysis must follow established documentation protocol. Adherence to this protocol provides a network of valuable information for documenting sample identification, tracking, and chain of custody.

GENERAL DOCUMENTATION PROCEDURES

Organization and concentration are the keys to completing the required documents efficiently and without error. Make certain that a suitable work area has been set aside with ample table and floor space for the processing of forms and the packaging of samples. This is especially important for large projects.

Forms and tags can be filled out in any order; however, past experience has shown that paperwork can be completed most efficiently and accurately if the sample identification matrix is completed before or in conjunction with the completion of the rest of the documentation. The recommended procedure is as follows:

1. Make or obtain a list of the samples to be packaged and shipped on the same day and the laboratories to be used.
2. Enter the case number, CRL number, matrix, sample numbers, laboratory, date sampled, and date shipped for each sample on the sample I.D. matrix.

Note: If portions of a given sample are to be shipped to different laboratories (e.g., for organic and inorganic analysis), two entry lines will be required for that sample number to accommodate the chain-of-custody record, airbill, and traffic report numbers corresponding to each portion of the sample.

3. Obtain the QC lot numbers of the prelabeled containers for each sample and enter them on the sample I.D. matrix.
4. Determine the number of shipping containers (coolers) required for the day's shipment. This will depend on the number of samples to be shipped, the number of containers per sample, the number of sample containers

that will fit in each cooler, and the number of laboratories to be used.

Note: A group of containers for a single sample should not be split between coolers unless the portions of the sample are to be sent to more than one laboratory for different types of analysis.

5. Complete an airbill for each laboratory address.

Note: Several coolers may be shipped to the same address under one airbill.

Shipment of medium and high concentration samples requires the use of a special airbill, including a shipper's certification for restricted articles.

6. Enter the airbill numbers on the sample I.D. matrix.
7. Assign a chain-of-custody record to each cooler and determine which sample containers will be shipped in each.

Note: More than one chain-of-custody record may be needed to accommodate the number of samples to be shipped in one cooler.

8. Assign chain-of-custody numbers to each sample by entering these numbers on the matrix.

Reminder: Portions of samples for organic and inorganic analysis will usually be sent to separate laboratories. Use one line on the sample I.D. matrix for the organic portion and another line for the inorganic portion.

9. If the samples are being shipped for routine analytical service (RAS), determine the number of traffic labels that will be needed for organics and inorganics.
10. Assign traffic report numbers from the labels to each sample and enter the numbers on the sample I.D. matrix.
11. Assign tag numbers to each sample container for each sample and enter the numbers on the sample I.D. matrix.
12. Complete one traffic report per laboratory each day (or SAS packing lists or CRL basic data sheets) based on the information provided on the matrix.

13. Complete sample tags according to the information provided on the sample I.D. matrix and the parameters of analysis. Place tags in groups by sample number.
14. Complete the chain-of-custody records based on the information provided on the sample I.D. matrix.
15. Assign two custody seals to each cooler. Enter the serial numbers of the seals in the "REMARKS" section of each chain-of-custody form and temporarily clip seals to the form.
16. Group all the paperwork associated with each cooler in a separate clip.
17. Obtain full signatures of the Sample Team Leader (STL) and initials of significant field team members (including yourself) on the sample tags and at the top of the chain-of-custody forms.
18. Prepare samples for shipment.

Following are step-by-step instructions for completing each form. Other items should be evident from the instructions.

SAMPLE IDENTIFICATION MATRIX (FIGURE 1)

1. Enter site name.
2. Enter project number.
3. Enter the case number.
4. Enter the CRL number.
5. Specify the sample matrix using the two- or three-digit codes listed below followed by the letter L, M, or H to indicate low, medium, or high concentration:
 - o SS--Surface Soil
 - o SB--Subsurface Soil
 - o SWO--Surface Water, Onsite
 - o SWC--Surface Water, Creek
 - o SDO--Sediment, Onsite
 - o SDC--Sediment, Creek
 - o GW--Groundwater
6. Enter the sample number.

7. Enter the organic traffic label number.
8. Enter the inorganic traffic label number.
9. Enter the chain-of-custody number.
10. Indicate the laboratory to be doing the analysis (abbreviations may be used as they are shown on the current laboratory list).
11. Enter the date the sample was taken: month, day, year (no hyphen or slash, e.g., 051284).
12. Enter the shipping date.
13. Enter the airbill number of the shipment.
14. List sample tag numbers corresponding to sample containers shipped under the traffic label number listed in either box 7 or 8.
15. List the QC lot numbers of the containers matching the tag numbers listed in Item 14.

Note: The date recorded on this form must be suitable for computer entry. Each entry must be flush left and must not exceed the number of digits allowed in each section. If portions of samples are to be sent to more than one laboratory for analysis, allow an entire line for each laboratory to accommodate for the additional traffic report, chain-of-custody, and airbill numbers.

SAMPLE TAG (FIGURE 2)

1. Enter the first six digits of the CRL sample identification.
2. Enter the last three digits of the CRL identification code.
3. Enter date of sampling.
4. Enter time of sampling (military time only).
5. Specify "grab" or "composite" sample with an "X."
6. Enter CH2M HILL sample identification code.
7. Obtain signature of sample team leader.

Form 1: Analytical Form

Project Code 1	Business No. 2	Month-Year-Year 3	Date 4	Preservative Yes <input type="checkbox"/> No <input type="checkbox"/> 5	ANALYSES 7	Remarks 10 10 11
Product Code				Tag No. 5-009071		
Business Location 6				Lab Sample No. 11		
Sampler's Signature 8				Analyses Table		

Analyses Table:

BOD Anions	
Solids (TSS) (TDS) (SS)	
COD, TOC, Nutrients	
Phenolics	
Mercury	
Metals	
Cyanide	
Oil and Grease	
Organics GC/MS	
Priority Pollutants	
Volatile Organics	
Pesticides	
Mutagenicity	
Bacteriology	

Form 2: Label

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
 REGION 5
 230 South Dearborn Street
 Chicago, Illinois 60604
 EPA

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FIGURE 2

Date: 10-31-88

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8. Indicate presence of preservative with an "X."
9. Specify all parameters for analysis with an "X" for each one.
- 10a. Indicate traffic label type and serial number (e.g., ITR number: MS 1534).
- 10b. Indicate case number and/or SAS number (e.g., Case No. 1234 and/or SAS No. 5678E).
11. Leave BLANK (for laboratory use only).
12. Enter any desired analyses not listed on menu (e.g., PCBs, ammonia, sulfide, etc.) and mark box with an "X."

INORGANIC TRAFFIC REPORT (FIGURE 3)

1. Enter assigned laboratory case number.
2. Enter assigned laboratory SAS number, if applicable.
3. Circle the code that describes the activity being performed.
- 4a. Enter site name.
- 4b. Enter city and state of site.
5. Enter EPA region number (e.g., V).
6. Enter sample team leader's company/office.
7. Enter sample team leader's name.
8. Enter laboratory name and address.
9. Enter name of laboratory contact.
10. Enter date samples were taken.
11. Enter "F" for Federal Express, date of shipment, and airbill number corresponding with the sample shipment.
12. Write on form if shipment is complete or is not complete.
13. Indicate sample description with a number (e.g., 1, 2, 3, 4, 5, 6, 7).

14. Specify sample concentration with an L, M, or H indicating contaminated concentration.
15. Check required analyses.
16. Specify special handling to notify laboratory if sample is a blank, M/S/D, field duplicate, or duplicate.
17. Enter CH2M HILL sample number.
18. Leave BLANK (for laboratory use only).

ORGANIC TRAFFIC REPORT (FIGURE 4)

1. Enter assigned laboratory case number.
2. Enter assigned laboratory SAS number, if applicable.
3. Circle the code that describes the activity being performed.
- 4a. Enter site name.
- 4b. Enter site city and state.
5. Enter EPA region number (e.g., V).
6. Enter sample team leader's name.
7. Enter sample team leader's office.
8. Enter laboratory name and address.
9. Enter name of laboratory contact.
10. Enter date samples were taken.
11. Enter "F" for Federal Express, indicate date of shipment, and indicate airbill number corresponding to sample shipment.
12. Write on form if shipment is complete or is not complete.
13. Specify sample description with a number (e.g., 1, 2, 3, 4, 5, 6, 7).
14. Specify the sample concentration with an L, M, or H.

U.S. ENVIRONMENTAL PROTECTION AGENCY
 CLP Sample Management Office
 P.O. Box 818 - Alexandria, Virginia 22313
 Phone: 703/557-2490 - FTS/557-2490

SAS Number

1

**SPECIAL ANALYTICAL SERVICE
 PACKING LIST**

Sampling Office: 2	Sampling Date(s): 5	Ship To: 8	For Lab Use Only Date Samples Rec'd: Received By:
Sampling Contact: 3 (name)	Date Shipped: 6	Attn: 9	
4 (phone)	Site Name/Code: 7		

Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		

For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

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 size.

FIGURE 5

REGION 5
230 South Dearborn Street
Chicago, Illinois 60604

PROJ. NO. (1)		PROJECT NAME (2)		NO. OF CONTAINERS (10)		REMARKS (12)	
SAMPLERS: (Signature) (3)							
STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION		(11)
(4)	(5)	(6)	(7)	(8)	(9)	(13)	
Relinquished by: (Signature)		Date / Time	Received by: (Signature)		Relinquished by: (Signature)		Date / Time
Relinquished by: (Signature)		Date / Time	Received by: (Signature)		Relinquished by: (Signature)		Date / Time
Relinquished by: (Signature)		Date / Time	Received for Laboratory by: (Signature)		Date / Time		Remarks (15)

Distribution: White — Accompanies Shipment; Pink — Coordinator Field File; Yellow — Laboratory File

5- 20445

FIGURE 6

3. Obtain full signature of sample team leader and signed initials of active team members (including paperwork person).
4. Enter last three digits of the CRL sample identification code.
5. List sampling dates for all samples.
6. List sampling times for all samples (military time only).
7. Indicate "grab" or "composite" sample with an "X."
8. List CH2M HILL sample numbers.
9. Enter number of containers per sample and container volume (e.g., 2-40 ml).
10. List analyses individually.
11. Enter column heading for traffic label number and list serial numbers for corresponding sample identification codes.
12. Write in the words "CASE #:" and enter the case number.
13. Enter column heading for "tag number" and list tag numbers for each sample container.
14. Obtain signature of sample team leader and carry out chain-of-custody procedures.
15. State carrier service and airbill number, lab service, and custody seal numbers.

NOTICE OF TRANSMITTAL (FIGURE 7)

1. Enter name of team leader.
2. Enter team leader's firm name.
3. Enter CH2M HILL project number.
4. Enter case number.
5. Enter date.
6. Enter number of samples shipped.

NOTICE OF TRANSMITTAL

DATE:

TO: CH2M HILL - REM/FIT Office, Reg. V-X (WI)
310 West Wisconsin Avenue, Suite 700
P.O. Box 2090
Milwaukee, Wisconsin 53201

Attn.: Shirley Stringer

FROM: ① / ②
(name) (firm)

CH2M HILL PROJECT #: ③

Enclosed are appropriate copies of the sample documentation

forms completed under Case # ④ for the
⑤, 19⑤ shipment of ⑥ ⑦
(qty) (matrix)
samples from the ⑧ site located in
⑨, ⑨.

GLT85/15

7. Enter matrix of samples.
8. Enter the site name in words.
9. Enter the location of the site (city, state).

FIELD LOG BOOK

All information pertinent to a field survey or sampling effort will be recorded in a log book or equivalent standard form. Each page or form will be consecutively numbered and will be at least 4-1/2 inches by 7 inches in size. All entries will be made in indelible ink or hard lead pencil and all corrections will consist of line-out deletions that are initialed and dated. As a minimum, entries in a log book will include the following:

- o Purpose of sampling
- o Location, description, and log photographs of the sampling point
- o Details of the sampling site (e.g., the elevation of the casing, casing diameter and depth, integrity of the casing, etc.)
- o Name and address of field contact
- o Documentation of procedures for preparation of reagents supplied which become an integral part of the sample (e.g., filters and absorbing reagents)
- o Identification of sampling crew members
- o Type of sample (e.g., groundwater, soil, sludge, wastewater)
- o Suspected waste composition
- o Number and volume of samples taken
- o Sampling methodology, including distinction between grab and composite samples
- o Sample preservation
- o Date and time of collection
- o Collector's sample identification numbers

- o Sample distribution and how transported (i.e., name of the laboratory and transporting agent)
- o References such as maps of the sample site
- o Any field measurements made (e.g., pH, specific conductance, temperature, and water depth)
- o Signature and date by the personnel responsible for observations
- o Decontamination procedures

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a log book or standardized form. However, records will contain sufficient information so that someone can reconstruct the sampling activity without relying on the sample collector's memory. The log book and standardized forms will be kept under strict chain-of-custody.

CORRECTIONS TO DOCUMENTATION

Unless prohibited by weather conditions, all original data recorded on traffic report forms, sample identification tags, chain-of-custody records, and receipt for sample forms will be written with waterproof ink. No accountable serialized documents are to be destroyed or thrown away, even if they are illegible or contain inaccuracies that require a replacement document.

If an error is made on an accountable document assigned to one individual, that individual shall make corrections by making a line through the error and entering the correct information. The erroneous information should not be obliterated. Any subsequent error discovered on an accountable document should be corrected by the person who made the entry. All subsequent corrections must be initialed and dated.

LABORATORY CUSTODY

Laboratory custody will conform to procedures established for the CLP. These procedures include:

- o Designation of a sample custodian
- o Correct completion by the custodian of the chain-of-custody record, sample tag, and laboratory

- request sheet (including documentation of sample condition upon receipt)
- o Laboratory sample tracking and documentation procedures
 - o Secure sample storage (of the appropriate environment--refrigerated, dry, etc.)
 - o Proper data logging and documentation procedures including custody of all original laboratory records

CENTRAL REGIONAL LABORATORY SAMPLE DATA REPORT (FIGURE 8)

The Central Regional Laboratory Sample Data Report is filled out by the CH2M HILL Sample Documentation Coordinator. A separate report is filled out for each laboratory that receives samples.

1. Enter the case number or SAS number.
2. Enter the site name.
3. Enter the laboratory name.
4. Enter the date shipped.
5. Enter the Superfund D.U. number.
6. Enter the EPA RPM.
7. Enter the CERCLIS number.
8. Enter the page numbers.
9. Enter the CRL numbers.
10. Enter the organic or inorganic traffic report number or the SAS packing list number.
11. Check the appropriate boxes for the analyses to be performed.

PACKING AND SHIPPING PROCEDURES

Sample packaging and shipping procedures are based on U.S. EPA Specifications and Department of Transportation (DOT) regulations (40 CFR). The procedures vary according to

THIS FORM IS TO BE USED FOR SAMPLES SENT TO CONTRACT ONLY

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FIGURE 8

sample concentration and matrix and are designed to provide optimum protection of samples and the public.

All samples will be shipped within 24 hours of collection. Shipping containers must be insulated, durable, and watertight. Bagged samples are to be cushioned within the shipping container with vermiculite packing material (zonolite). To prevent contamination of samples, all containers regardless of size and type must be placed inside sealed plastic bags before being packed in vermiculite or zonolite. Preformed poly-foam cooler liners may be used for shipment of low-concentration samples only.

Following shipment, airbill numbers must be called in to the SMO and to the sample documentation coordinator.

LOW-CONCENTRATION SAMPLES

1. Prepare coolers for shipment.
 - o Tape drains shut.
 - o Affix "This Side Up" labels on all four sides and "Fragile" labels on at least two sides of each cooler.
 - o Place mailing label with laboratory address on top of coolers.
 - o Fill bottom of coolers with about 3 inches of vermiculite or use performed poly-foam liner.
 - o Place appropriate traffic reports, SAS packing lists, or regional field sheets and chain-of-custody records with corresponding custody seals on top of each cooler.
2. Arrange decontaminated sample containers in groups by sample number.
3. Mark volume levels on bottles with a grease pencil.
4. Secure appropriate sample tags around lids of containers with string or wire.
5. Secure container lids with strapping tape.
6. Arrange containers in front of assigned coolers.

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7. Affix appropriate adhesive labels from assigned traffic report to each container. Protect with clear label protection tape.
8. Seal each container within a separate plastic bag.
9. Arrange containers in coolers so that they do not touch.
10. If ice is required to preserve the samples, cubes should be repackaged in double zip-loc bags and placed on and around the containers (especially on VOA vials).
11. Fill remaining spaces with vermiculite (or place poly-foam liner cover on top of samples).
12. Sign chain-of-custody form (or obtain signature) and indicate the time and date it was relinquished to Federal Express.
13. Separate copies of forms. Seal proper copies within a large zip-loc bag and tape to inside lid of cooler. Distribute remaining copies as indicated in the following sections.
14. Close lid and latch.
15. Carefully peel custody seals from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape.
16. Tape cooler shut on both ends, making several complete revolutions with strapping tape. Do not cover custody seals (see Figure 9).
17. Relinquish to Federal Express. Place airbill receipt inside the mailing envelope and send to the sample documentation coordinator along with the other documentation.
18. Telephone the SMO in Alexandria, Virginia.

Note: This step should be omitted for samples sent to the CRL).

Ms. Leslie Braun (subject to change)
(703) 557-2490

Provide the following information:

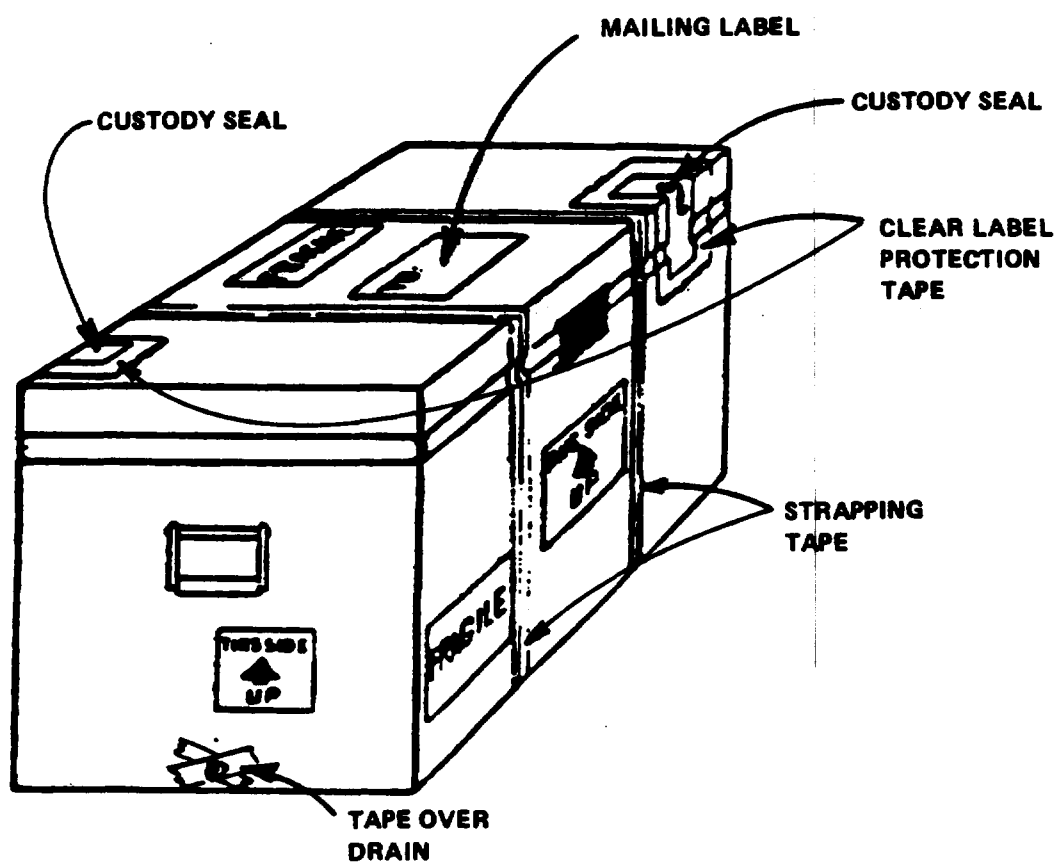


FIGURE 9

- o Your name
- o Project name
- o Case number
- o Number of samples sent to each laboratory for analysis
- o Airbill numbers

This must be done IMMEDIATELY following sample shipment. If the SMO is closed at that time, call in the information first thing the next day.

MEDIUM- AND HIGH-CONCENTRATION SAMPLES:

Medium- and high-concentration samples are packaged using the same techniques used to package low-concentration samples, with several additional restrictions. First, a special airbill including a Shipper's Certification for Restricted Articles is required (Figures 10 and 11). Second, "Flammable Liquid N.O.S." or "Flammable Solid N.O.S." labels must be placed on at least two sides of the cooler. Third, sample containers are packaged in metal cans with lids before being placed in the cooler, as indicated below.

- o Place approximately one-half inch of vermiculite in the bottom of the can.
- o Position the sample jar in the zip-loc bag so that the sample tags can be read through the plastic bag.
- o Place the jar in the can and fill the remaining volume with vermiculite.
- o Close the can and secure the lid with metal clips.
- o Write the traffic report number on the lid.
- o Place "This Side Up" and "Flammable Liquid N.O.S." (or "Flammable Solid N.O.S.") labels on the can.
- o Place the cans in the cooler.

AIRCRAFT NUMBER 395461054		SHIPPER'S CERTIFICATION FOR RESTRICTED ARTICLES (TYPE OR PRINT)									
SHIPPER'S NAME: _____ CLASSIFICATION: _____											
ADDITIONAL DESCRIPTION REQUIREMENTS FOR RADIOACTIVE MATERIALS (SEE BACK)	<table border="1"> <tr> <td style="height: 40px;"></td> <td style="height: 40px;"></td> <td style="height: 40px;"></td> <td style="height: 40px;"></td> <td style="height: 40px;"></td> <td style="height: 40px;"></td> </tr> </table>										
THIS SHIPMENT IS WITHIN THE LIMITATIONS PRESCRIBED FOR <table border="1"> <tr> <td>PASSENGER AIRCRAFT</td> <td>CARGO AIRCRAFT ONLY</td> <td>(DELETE-NONAPPLICABLE)</td> </tr> </table>					PASSENGER AIRCRAFT	CARGO AIRCRAFT ONLY	(DELETE-NONAPPLICABLE)				
PASSENGER AIRCRAFT	CARGO AIRCRAFT ONLY	(DELETE-NONAPPLICABLE)									
IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS OR TREATMENT.											
I HEREBY CERTIFY THAT THE CONTENTS OF THIS CONSIGNMENT ARE FULLY AND ACCURATELY DESCRIBED ABOVE BY PROPER SHIPPING NAME AND ARE CLASSIFIED, PACKED, MARKED, AND LABELED, AND IN PROPER CONDITION FOR CARRIAGE BY AIR ACCORDING TO APPLICABLE NATIONAL GOVERNMENTAL REGULATIONS.											

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FIGURE 11

DISTRIBUTION OF COMPLETED DOCUMENTS

Final disposition of the completed documents is as follows:

- o Shipped with Samples:
 - Chain-of-custody form, white original
 - Traffic report forms, white and yellow copies
 - SAS packing list, pink and gold copies
 - Sample tags
- o Retained by Project Manager:
 - Sample identification matrix
 - Field log books (at completion of project)
- o Sent to CH2M HILL Documentation Coordinator:
 - Chain-of-custody form, pink and yellow copies
 - Traffic report forms, white original and pink copies
 - SAS packing list, white original and yellow copy
 - Notice of transmittal

SPECIAL INSTRUCTIONS FOR SHIPPING SAMPLES BY FEDERAL EXPRESS

1. Label cooler as hazardous shipment.
 - o Write shipper's address on outside of cooler. If address is stenciled on, just write "shipper" above it.
 - o Write or affix sticker saying "This Side Up" on two adjacent sides.
 - o Write or affix sticker saying "ORM-E" with box around it on two adjacent sides. Below ORM-E, write NA#9188.
 - o Label cooler with "Hazardous Substance, N.O.S." and "liquid" or "solid," as applicable.
2. Complete the special shipping bill for restricted articles (Figures 10 and 11).

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- o Under Proper Shipping Name, write "Hazardous Substance, N.O.S." and "liquid" or "solid," as applicable.
- o Under Class, write "ORM-E."
- o Under Identification No., write NA No. 9188.

GLT821/20

Appendix G
PROPOSED NPDES DISCHARGE LIMITS

GLT821/12-3

DEPARTMENT OF ENVIRONMENTAL MANAGEMENT

OFFICE MEMORANDUM

DATE: April 10, 1987

TO: John Buck

THRU: Joseph Krieger *JK*
Larry Kane
Joseph Stallsmith
Jane Magee
Glenn Pratt

FROM: Brad Gavin *BG*

SUBJECT: Proposed Design and NPDES Permit Limits for the North Side Landfill

Attached are some design and permit limits for the North Side Landfill. The limits for the parameters copper, lead, zinc, hexavalent chromium, and cyanide, were determined using EPA criteria for toxicity to aquatic life. The limits for the parameters arsenic, tetrachloroethylene, trichloroethylene, methylene chloride, benzene, and phenanthrene were determined using EPA criteria for protection of human health from the carcinogenic effects of these compounds at the 10^{-6} risk level for people consuming aquatic organisms only. The limit for 4-chloro-3-methylphenol is one-tenth the 96-hour LC50 for the compound. The limits for TSS, oil & grease, chloride, and iron were determined using available toxicity data for the compounds and best professional judgment. The flow used to determine all of the limits was the $Q_{7,10}$ flow of Finley Creek, which, given the limited drainage area is assumed to be 0.0 cfs.

Northside Landfill
Proposed Permit Limits
4/09/87

<u>Parameter</u>	<u>Discharge Limits</u>		<u>Monitoring Requirements</u>		<u>Design Limits</u>
	<u>Monthly</u>	<u>Daily</u>	<u>Frequency</u>	<u>Type</u>	<u>Long Term</u>
	<u>Average</u>	<u>Maximum</u>			<u>Average</u>
	mg/l				mg/l
TSS	10	20	Twice Weekly	24-Hr. Comp.	--
Oil and Grease	--	10	Twice Weekly	Grab	--
Chloride	--	1,500	Twice Weekly	24-Hr. Comp.	--
Total Recoverable Copper	0.015	0.023	Twice Weekly	24-Hr. Comp.	0.011
Total Recoverable Lead	0.009	0.014	Twice Weekly	24-Hr. Comp.	0.0066
Total Recoverable Zinc	0.098	0.17	Twice Weekly	24-Hr. Comp.	0.0658
Total Recoverable					
Hexavalent Chromium	0.006	0.009	Twice Weekly	24-Hr. Comp.	0.0042
Total Cyanide	0.006	0.01	Twice Weekly	24-Hr. Comp.	0.0047
Total Iron	--	1.00	Twice Weekly	24-Hr. Comp.	--
Total Arsenic*	0.0000175	--	Twice Weekly	24-Hr. Comp.	--
4-Chloro-3-Methylphenol	--	0.001	Twice Monthly	Grab	--
Tetrachloroethylene	0.009	--	Twice Monthly	Grab	--
Trichloroethylene	0.081	--	Twice Monthly	Grab	--
Methylene Chloride	0.016	--	Twice Monthly	Grab	--
Benzene	0.04	--	Twice Monthly	Grab	--
Phenanthrene**	0.00003	--	Twice Monthly	24-Hr. Comp.	--
Chronic Toxicity***	Monitor Only		Quarterly	24-Hr. Comp.	--

*The detection level of arsenic is 0.001 mg/l while the monthly average limit is 0.0000175 mg/l. If the permittee never exceeds 0.001 mg/l of arsenic in their effluent the permittee will be in compliance with the permit.

**The detection level of phenanthrene is 0.0054 mg/l while the monthly average limit is 0.00003 mg/l. If the permittee never exceeds 0.0054 mg/l of phenanthrene in the effluent the permittee will be in compliance with the permit.

***The permittee shall monitor chronic toxicity of the effluent using the fathead minnow growth test and the ceriodaphnia life cycle test quarterly for a period of one year. If after that sampling has been completed and the effluent has been found not to be chronically toxic the permittee will be allowed to reduce the toxicity monitoring to yearly.

INDIANAPOLIS

OFFICE MEMORANDUM

DATE: May 27, 1987

TO: John Buck *JB* THRU: Joseph Krieger *JK* 5-27
Larry Kane *LK*
Joseph Stallsmith *JS* 5-28 87
Jane Magee *JM*
Glenn Pratt

FROM: Brad Gavin

SUBJECT: Additional NPDES Permit Requirements for the North Side Landfill

On April 10, 1987 I sent a memo regarding some proposed permit limits for the North Side Landfill. The memo never made it through channels and hasn't been located. Attached find a copy of that earlier memo and a list of proposed permit requirements for some additional pollutants. The limits for the parameters chloroform, and 1,1-dichloroethylene were determined using EPA criteria for protection of human health from the carcinogenic effects of these compounds at the 10^{-6} risk level for people consuming aquatic organisms only. These limits should adequately protect the people which receive their drinking water from the Eagle Creek Reservoir from the carcinogenic properties of these compounds. The limit for phenol is one-tenth the 96-hour LC50 for the compound. Monitoring for cis-1,2-dichloroethylene will be required since it has been frequently detected in the receiving stream downstream of the North Side Landfill. A GC/MS scan will be required quarterly to determine if unpermitted parameters are being discharged in harmful quantities. The flow used to determine all of the limits was the $Q_{7,10}$ flow of Finley Creek, which, given the limited drainage area is assumed to be 0.0 cfs.

Northside Landfill
Proposed Permit Limits
5/27/87

Parameter	Discharge Limits		Monitoring Requirements	
	Monthly Average	Daily Maximum mg/l	Frequency	Type
Phenol	--	0.57	Twice Weekly	24-Hr. Comp.
1,1-Dichloroethylene	0.002	--	Twice Monthly	Grab
Chloroform	0.016	--	Twice Monthly	Grab
Cis-1,2-Dichloroethylene	Monitor Only		Twice Monthly	Grab
GC/MS Scan	Monitor Only		Quarterly	As required by test method

DEPARTMENT OF ENVIRONMENTAL MANAGEMENT

INDIANAPOLIS

OFFICE MEMORANDUM

DATE: June 7, 1988

TO: John Buck *SPB 7/12/88*
Office of Environmental ResponseTHRU: Joe Krieger *JK*
Lon Brumfield *LB 4/7*
Joe Stallsmith *JS 7-1*

FROM: Brad Gavin

SUBJECT: Northside/ECC Influent Characterization Study

I am sending you some revised limitations for the Northside landfill discharge. Also, as we discussed on the telephone, I am also sending a copy of the limitations to Al Lao of the Facility Planning Section to review the influent data and the proposed treatment system to verify that it will be able to meet the proposed NPDES Permit limits. We are asking that he comment to you directly through channels, with a copy sent to us, by June 30, 1988.

North Side Landfill

Parameter	Proposed Limits	
	Average	Maximum
	mg/l	
Arsenic[1]	0.0002	0.0003
Hex. Chromium	0.008	0.018
Copper	0.021	0.048
Cyanide[1]	0.005	0.009
Iron	0.71	1.6
Lead	0.009	0.02
Zinc	0.184	0.429
Chloride	160	373
4-Chloro-3-Methylphenol[1]	0.0022	0.0044
Tetrachloroethene[2]	0.022	0.056
Trichloroethene[2]	0.021	0.054
Methylene Chloride[2]	0.04	0.089
Benzene[2]	0.037	0.136
Phenanthrene[1]	0.002	0.004
Phenol[2]	0.015	0.026
1,1-Dichloroethene	0.005	0.012
Chloroform[2]	0.021	0.046
Cis-1,2-Dichloroethylene	Monitor Only	
Vinyl Chloride[2]	0.104	0.268
Chloroethane[2]	0.104	0.268
CBOD ₅	10	20
TSS	12	24
Phosphorus	1[3]	
Dissolved Oxygen	6.0 minimum daily average	
Ammonia[4]		
Summer	1.5	3.0
Winter	3.0	6.0

[1] The limitations for the above noted parameters are based on the quantification limits for those parameters. The limitations based on Water Quality Standards are given below. If more precise methods of analysis are approved the permit may be modified to recognize the lower detectable values.

Arsenic	0.0001	0.0003
Cyanide	0.004	0.009
4-Chloro-3-Methylphenol	0.00005	0.00012
Phenanthrene	0.0002	0.0005

[2] The limitations for the above noted parameters are based on the Best Professional Judgement (BPJ) of BAT for this discharge.

[3] Or the percent removal required by 327 IAC 5-10, whichever is more stringent.

Phosphorus in Raw Sewage

Removal Required

Greater than or equal to 4	80%
less than 4, greater than or equal to 3	75%
less than 3, greater than or equal to 2	70%
less than 2, greater than or equal to 1	65%
less than 1	60%

[4] The limitations for ammonia above are based on the available treatment technology. The limitations protective of water quality are 1.1 mg/l monthly average and 2.3 mg/l daily maximum in the summer, and 1.6 mg/l monthly average and 3.3 mg/l daily maximum in the winter. If economically viable treatment technology becomes available to treat ammonia to lower levels this permit will be modified to reflect the new treatability limits.